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TECHNICAL REPORT 4067

INVESTIGATION OF POTASSIUM DINITROBENZOFUROXAN (KDNBF) TO PROVIDE DATA NECESSARY FOR THE PREPARATION OF A MILITARY SPECIFICATION

T. S. COSTAIN

NOVEMBER 1970

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Explosives Laboratory
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Picatinny Arsenal
Dover, N. J.

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OBJECTIVES

1. To develop recommendations for a manufacturing procedure for KDNBF.

2. To evaluate commercially procured KDNBF, and compare it with KDNBF of known purity and properties.

3. To interrelate the physical, chemical, and explosive properties of KDNBF from all sources so as to define the critical parameters which must be specified to assure close control of the final product.

ABSTRACT

Under a program funded by the Nuclear Engineering Directorate, the Explosives Laboratory of Feltman Research Laboratories, Picatinny Arsenal, has defined the critical properties of KDNBF (commonly known as potassium dinitrobenzofuroxan, but more accurately designated as potassium 4, 6-dinitro-7-hydroxy-7-hydrobenzofuroxan), established a self-checking testing procedure, and set tolerances which can be used as a basis for a rigorous military specification. KDNBF's of both particularly high and low purity and quality were tested concurrently with KDNBF's obtained from three commercial sources to provide a broad diversity of data. The functioning of KDNBF was defined from pressure-time traces obtained from firings in a 1-ml (milliliter) pressure bomb, and it was related to purity as determined by nuclear magnetic resonance, X-ray diffraction, and elemental analysis. The thermal stability, as measured by differential thermal analysis and gas evolution at 120°C, also was related to KDNBF purity and function. A specific test based on solubility in methanol was devised to detect contamination of KDNBF by a hydrolysis decomposition product and, in addition, storage under absolute methanol in sealed containers was recommended. Of secondary importance were tests related to color, form, granulation, and bulk density, which were used mainly to describe a form of KDNBF suitable for use in automatic loading machinery. In the course of the study the manufacturing parameters for KDNBF were systematically varied so that a judgment on the effects and relative importance of raw materials, intermediate compounds, and unit processes could be reached. A recommended process of manufacture is presented.

Although not a part of the recommendations for a specification, values for the various KDNBF's were obtained in the Picatinny Arsenal Impact Sensitivity Test and the Picatinny Arsenal Explosion Temperature Test. The compatibility of KDNBF with several binder resins, a glass-filled polycarbonate, and a fluorinated silicone rubber also was determined.

CONCLUSIONS

The recommended process of manufacture is given as part of the Draft Purcahse Description (Appendix A). Examination of Figures 1A and 1B will show the following points:

1. In the production of intermediate compounds (i.e., benzofuroxan, dinitrobenzofuroxan) the process of manufacture is as important as the purity of the raw materials (within the limits of purity tested).

2. However, if DNBF of reasonable purity is used, an acceptable KDNBF can be produced.

3. The most critical point in the manufacturing process is the actual precipitation of KDNBF. Variations at earlier points can be compensated for by simple recrystallization of the DNBF.

The properties of the three commercial KDNBF's were very similar to one another. When tested as outlined in Appendix A, the results obtained show in general that the commercial KDNBF's lie between the purest material (i.e., KDNBF-45) and crude KDNBF. All of them would pass the criteria established.

To summarize the testing program, it may be said that the essential tests are function (1-ml bomb), NMR, DTA, and contamination by methanol-soluble material. But this is a bare skeleton structure, and all the other recommended tests are required as cross-checks if near 100 percent assurance is desired. The content of the Scope of Work (Appendix C) would indicate that KDNBF is used in a very critical application and that more than ordinary precaution must be observed.

RECOMMENDATIONS

Possible areas for future studies would include:

1. Investigation of long-term storage stability of KDNBF under anhydrous conditions.

2. Determination of the mechanism of decomposition by hydrolysis and of the structure of the decomposition product or products.

3. Investigation of other salts of DNBF and of KDNBF itself for possible use as initiating explosives.

INTRODUCTION

KDNBF has found use in various explosive-actuated devices. Its desirable properties include easy ignitability, low "Z" factor, fast response, and nonconductive ash.

The compound was first prepared in 1899 by von P. Drost. Recently its structure was defined by N. E. Brown, et al (Ref 1) and some of its characteristics were determined by A. Anzalone, et al (Ref 2).

Up to now, KDNBF has been procured as a component of commercially produced devices, with no U. S. Government specification to define the properties of the material itself. Increased use of KDNBF has, however, given rise to a need for a comprehensive specification to insure that devices obtained in future procurements contain high quality KDNBF.

Therefore a proposal (Appendix 2) was prepared by the Explosives Laboratory and forwarded to the Nuclear Engineering Directorate. The Nike-X Engineering Division responded with a Scope of Work (Appendix 3) which formed the basis for the effort reported in this study.

RESULTS[1]

Process of Manufacture

The process of manufacture developed starts with orthonitroaniline which is oxidized to benzofuroxan. The benzofuroxan is nitrated to dinitrobenzofuroxan, which in turn is reacted with potassium carbonate or bicarbonate to form the final product, potassium dinitrobenzofuroxan. Details of this process are given in Experimental Procedures.

Melting Point Determination

The melting points of the various raw materials and intermediate compounds are given in Table 1 and Figure 1. The instrument used

[1] Most of the materials mentioned in this section are identified with a numeral, or a numeral and a letter, following the initials of the chemical name (e.g., KDNBF-45; DNBF-60E) the numeral being the page number of Logbook 764-105. The three commercially procured KDNBF's are suffixed by the letters X, Y, or X, respectively. Reference to Figures 1A and B will be helpful in identifying materials and their relationship to other materials.

was standardized against sulfanilamide, sulfapyridine, and vanillin melting-point standards provided by the manufacturer (Arthur H. Thomas Co.).

Elemental Analysis

The analysis of the materials for their constituent elements was conducted by a commercial laboratory that specializes in microchemical analyses. The results are given in Table 2.

Differential Thermal Analysis

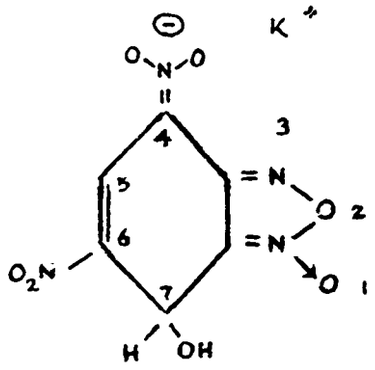
The DTA curve of KDNBF in a helium atmosphere exhibits only one major exotherm in the region of 210° - 220°C. A possible secondary exotherm near 300°C was not considered significant. In Appendix A, Figure A7, the DTA of KDNBF-45 (auto-loading grade, Type I) is given. Figures A8 and A9 (Appendix A) are, respectively, for commercial KDNBF (Company Z) and KDNBF-78 (crude). In addition, KDNBF-66B (auto-loading grade Type II) showed an exotherm at 209°C; while the other two commercial KDNBF's, X and Y, showed exotherms at 220°C, respectively.

Ultraviolet and Infrared Spectroscopy

The UV and IR spectra of KDNBF and its decomposition product were investigated. However no differences of a useful nature were observed; and since, besides, other methods of identification proved more productive, no further work on UV and IR was done.

Nuclear Magnetic Resonance

The NMR spectrum of KDNBF was determined on a Varian T-60 Spectrometer and run in a 15% solution (W/V) of deuterated dimethylsulfoxide using tetramethylsilane as an internal reference. It exhibited 2 doublets centered at 5.87 ppm(parts per million) from TMS with a coupling constant of 8 cps, and a singlet at 8.63 ppm (Fig A10, Appendix A). This is consistent with the Meisenheimer structure advanced by Brown et al (Ref 1) for hydrolyzed 4, 6-dinitrobenzofuroxan. Consequently, the Meisenheimer structure for KDNBF should be:



The NMR resonance line at 8.63 ppm is attributed to the ring proton at Position 5. Inasmuch as the OH proton at Position 7 is deshielded, it should appear as the doublet at 6.20 ppm, and the remaining proton at Position 7 should appear upfield. To show this, the KDNBF solution was shaken with a small amount of D_2O. This caused considerable collapse of the doublet at 6.20 ppm and obvious singlet formation at 5.87 ppm, indicating correct assignment. The water line at about 3.3 ppm was accordingly increased (Fig 2). The small singlet at 5.87 ppm present in the KDNBF spectra before and after treatment with D_2O cannot be accounted for, but it probably indicates a small quantity of impurities.

Spectra were obtained of samples of KDNBF labeled KDNBF-45 (Appendix A, Fig A10); KDNBF-Z (Appendix A, Fig A11); KDNBF-Y (Fig 3); KDNBF-X (Fig 4); and KDNBF-78 (Appendix A, Fig A12). Of these, the first three (45, Z, and Y) produced spectra with fairly sharply resolved resonance lines. The spectra of samples A and 78 were not as well-defined, possibly indicating the presence of some impurity that caused broadening of the doublets.

The fact that KDNBF-X had been stored for more than three weeks under a solvent suspected of containing water could account for the lower apparent purity.

X-Ray Diffraction

X-ray diffraction patterns were obtained for KDNBF-45 (autoloading grade, Type I, Appendix A, Fig A13); KDNBF-66B (autoloading grade, Type II); and KDNBF-X (commercial, company X). The patterns from all three were identical, indicating that the elemental and crystalline structure of these three very different-appearing materials (see Fig A3, A4 and A5, Appendix A) was the same. The differences are merely in crystal habit and color.

Contamination by Methanol-Soluble Substances

In the course of this study it was determined that KDNBF reacts with water to produce an undesirable decomposition product (DP). The results of elemental analysis (Table 2) indicate that storage in contact with water for five years (DP-31 SLR) causes more complete degradation than does immersion in boiling water for six hours (DP-52C). The concentration of DP in the supernatant liquor (alcohol with at least 5 percent water) over KDNBF of Lot SMUPA-7172, which had been in storage for five years, was between 4 and 5 percent.

Materials from companies Y and Z were obtained with absolute methanol as the supernatant liquor. The material received from company X was covered with another liquid which, it is suspected, was 3A alcohol. All samples were, when inspected, at least three weeks old. All supernatant liquid was filtered off and replaced with fresh methanol. Two months later, when it was discovered that company X's liquid differed from methanol, the rewetted samples of KDNBF-X were filtered for recovery of the methanol supernatant liquid that had been used to replace the original liquor.

Tests were conducted to determine the approximate solubility in methanol of both the decomposition product (DP) and KDNBF. The room temperature (22°C) solubility of DP was 357 mg per 100 ml of methanol, while that of KDNBF was only 37 mg. The saturated solution of DP was almost opaque and burnt-amber in color.

The original supernatant methanol from samples KDNBF-Y and KDNBF-Z and the methanol from the rewetted sample of KDNBF-X were tested to determine the contamination by methanol-soluble substances. The results obtained were:

Solute content of supernatant liquor at room temperature

	KDNBF-X	KDNBF-Y	KDNBF-Z
Grams per 100 ml methanol	0.145	0.110	0.090
Grams per 100 g KDNBF	0.50	0.30	0.47

Vacuum Stability

The results of testing several samples of KDNBF in the vacuum stability test at several temperatures are as follows: (The entry "11+" indicates that the capacity of the apparatus was exceeded.)

Milliliters of gas evolved per gram of KDNBF in 40 hours at:

Sample KDNBF	110°C	120°C	130°C
45 (purest)		0.73	
78 (crude)	11+		
X	0.49	0.69	3.01
Y	0.41	0.56	1.50
Z	0.73	0.74	3.74

The above results correlate well with those obtained in the tests for contamination by methanol-soluble substances conducted on the commercial materials.

The results of testing KDNBF intimately mixed with the indicated materials in the vacuum stability test at 120°C are as follows:

Mixture	Milliliters of gas evolved per gram of KDNBF at 120°C in 40 hours
50/50 KDNBF-X/plastic, polycarbonate, glass-reinforced, Type III, Mil P81390	0.62
50/50 KDNBF-X/Silastic, LS 53	0.35

The following results were obtained in vacuum stability tests of spotting mixtures of KDNBF in lacquer.

Mixture (containing KDNBF, SMUPA-7172)	Milliliters of gas evolved per gram of sample, at 120°C in 40 hours
100/0 KDNBF	0.58
90/10 KDNBF/Ethyl Cellulose (48 percent Ethoxy)	0.45
90/10 KDNBF/Nitrocellulose (12 percent N)	9.67
90/10 KDNBF/Zytel 63 (nylon resin)	3.22
90/10 KDNBF/Butvar B72A (polyvinylbutyral)	0.25

Explosion Temperature Test

Material Tested	Explosion Temperature (5-second point)
KDNBF-45	247°C
KDNBF-78	226°C
KDNBF-X	252°C
KDNBF-Y	259°C
KDNBF-Z	253°C

Picatinny Arsenal Impact Sensitivity Test

Material Tested	Average Weight of Samples (gram)	Impact Test Value (inches) using 2-kg wt
KDNBF-45	.006	7
KDNBF-66B	.009	7
KDNBF-X	.009	9
KDNBF-Y	.009	5
KDNBF-Z	.013	8

These results would indicate that KDNBF is, at least in this test, less sensitive to initiation than dextrinated lead azide whose impact test value is listed in the literature as 5 inches. KDNBF would be classified as an Initiating Explosive under existing ICC regulations.

Bulk Density and Sieve Analysis[1]

The granulation of the three commercial KDNBF's was determined using a modified version of the usual wet sieving method. Since KDNBF is to some extent soluble in alcohols and water, the amount of fluid used had to be reduced to a minimum. It was, therefore, convenient and useful to combine the determination of bulk density with the sieve analysis. Also, an extremely accurate method was not warranted. The results obtained follow:

Sample KDNBF	Bulk Density grams/ml	Percent on U. S. Sieve No: 50	100	200	270	PAN	Lost
X	.28	1.5	1.5	67.0	26.0	4.5	-
Y	.36	0.5	0.0	9.5	72.0	15.0	3.0
Z	.15	1.0	3.5	70.0	12.5	12.5	0.5

Pressure-Time Relationships in a 1-ml Bomb

The pressure-time curves for several KDNBF's were determined in a 1-milliliter volume pressure bomb. A general description of this test in included in Appendix A. To be more specific, this test was performed in a bomb with a measured volume of 1.08 ml. A Kistler 701-H, 10 to 10,000 psi transducer, S/N 22831, was used as the pressure-sensing device. A constant 1.00 ± 0.01-ampere current was supplied to the bridgewire from a Universal Pulser (Franklin Institute). The bridgewires were spotted with 90/10 KDNBF/Butvar B72-A resin.

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[1] See Appendix A for experimental procedure

Individual test results are given in Table 3. Plots of these data on probability graph paper are shown in Figure 5, 6, and 7.

KDNBF (samples numbered)	Time to Ignition[1] millisec	Time to 90% Maximum Pressure[1], msec	Maximum Pressure, psi
45 (25 to 32)	1.16	2.04	52.3
78 (35 to 44, less 40)	1.07	2.16	42.7
X (9 to 16, less 10)	1.08	2.60	43.2
Y (1 to 7)	1.08	2.56	38.8
Z (17 to 23)	1.07	3.11	44.4

One additional series of pressure bomb tests were performed, but the pressure values are not presented here because at the time the current through the bridgewire could not be controlled with enough precision. However, the time-to-ignition values vs current were grouped and averaged, and the results are presented in Figure 8 and tabulated below:

Current, amperes	Individual ignition time (milliseconds) for: 90/10 KDNBF-X/nitrocellulose	90/10 KDNBF-X/Butvar B72-A
0.85	1.75, 1.75, 1.45	1.30, 1.30
0.925		
1.00	1.15, 1.28, 1.40	
1.15	1.05, 0.85, 0.90	0.85, 0.85, 0.90

[1] Both "time to ignition" and "time to 90 percent of maximum pressure" were measured from the start of the flow of current through the bridgewire

DISCUSSION OF RESULTS

Since the most important objective of this study was to relate the functioning characteristics of KDNBF to its quality or purity, the 1 -ml pressure bomb tests should be ranked highest in importance. They measured the three parameters most likely to affect the performance of KDNBF in an explosive-actuated device (e.g., squib switch). The results show that KDNBF's of different chemical and physical characteristics are nearly identical in functioning characteristics.

When the ignition times (Fig 6) and the maximum pressures (Fig 5) are plotted on probability graph paper, the results fall in both cases on a straight line (except, of course, for the "tails"). This shows that any variations can be attributed to normal random scattering of the data. The plot of "Times to 90 percent of maximum pressure" (Fig 7) does not fall on a straight line, the curve being skewed on both ends. Examination of the individual tests in Table 3 or even the averaged values previously given shows that this skewing is due to the excessively fast burning of KDNBF-45 and the excessively slow burning of KDNBF-Z.

Ordinarily, an explosive-actuated device is designed to function at some point between ignition time and maximum pressure buildup. Generally, functioning is projected to occur in the range of 25 to 50 percent of maximum pressure. Thus it may be that the slower pressure buildup of KDNBF-Z will have little effect in an actual item. Since, however, we cannot be certain of this contingency, the limits given in the appended Draft Purchase Description have been chosen so that the performance data listed for KDNBF-Z represent only borderline values.

The fact that KDNBF-78 (impure and unsuitable for use) also passed the functioning test only emphasizes the need for supplemental tests. The system developed and presented here in the form of a Draft Purchase Description (Appendix A) constitutes a rigorous basis for a specification. It is intended to allow acceptance of KDNBF of the type presently produced by commercial sources. But it goes a step further in specifying a new automatic-loading grade of KDNBF as represented by KDNBF-45. Present commercial KDNBF is usable for the limited application of hand-loading lines, but the fine flaky form of the crystals make it unsuitable for use in automatic loading

machinery. However, since present applications call for hand-loading, there is no need to require the use of the automatic-loading grade exclusively.

Some of the requirements are noncritical and are intended only for monitoring successive lots of product to insure that reproducible methods are used. These tests are: Bulk density, color, sieve analysis and elemental analysis.

Since all the limits for the automatic loading grade would not be set at this time (provision being made for submission of a preproduction sample), some of the above requirements could assume critical status for a particular application (e.g., bulk density) and should be so specified in the purchase contract.

Another pair of tests, differential thermal analysis and vacuum stability, measure the same property, i.e., thermal stability, but DTA measures it with greater precision and over a much wider temperature range. Both tests serve to eliminate material of low purity as typified by KDNBF-78. This apparent redundancy is intentional to provide a cross-check. Another thermal stability test, the explosion temperature test was conducted but it was omitted from Appendix A because it is difficult and expensive to perform properly, and it is easy to obtain any desired result if performed improperly.

An important and critical test is the one for contamination by methanol-soluble substances. It is designed to detect any impurity resulting from the hydrolysis of KDNBF. Significant amounts of this decomposition product are formed when KDNBF is recrystallized from water, a normal manufacturing procedure, and also in long-term storage in the presence of even small (i.e., 5 percent) quantities of water. Denatured 3A alcohol contains water, and even absolute alcohols will pick up water from the atmosphere unless kept in sealed containers. Freshly prepared KDNBF which had been washed with methanol and dried should be virtually free of the decomposition product, and if it is loaded into hermetically sealed items, it will be safe. However, KDNBF in storage is in potential danger of deterioration. After five years of storage, one batch of KDNBF, lot SMUPA 7172, was dried without washing. The resultant hard cake had to be destroyed since it was completely unusable.

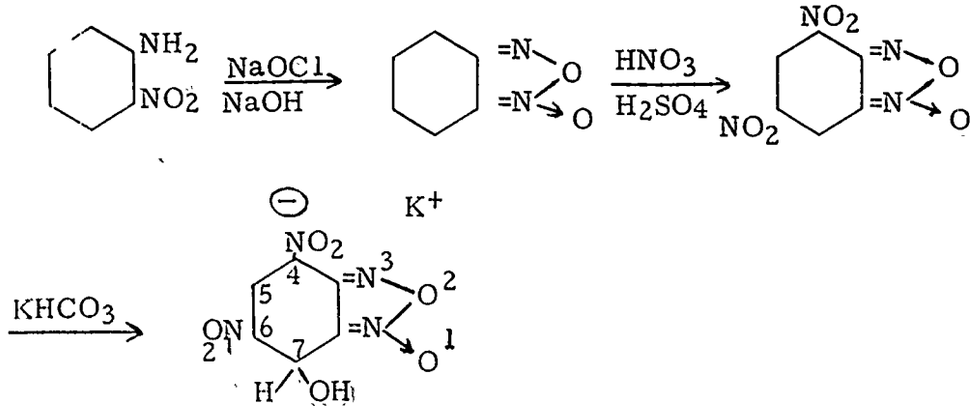
Other batches of SMUPA Lot 7172, washed thoroughly before drying, yielded a product not unlike commercial KDNBF. Earlier tests (Ref 4) appeared to indicate that KDNBF rehabilitated by washing out any decomposition product, gave acceptable results in a functioning test. However, long-term effects could not be determined.

The final pair of tests suggested are designed to identify KDNBF as a composition of matter. The first of these tests, nuclear magnetic resonance, is a pertinent and valuable one for KDNBF analysis because of its simple proton structure. The resultant spectra can be interpreted, and even a qualitative judgment of the purity of the KDNBF can be made by visual comparison with standard spectra. The second test was X-ray diffraction, which was included at the request of the Quality Assurance Division.

EXPERIMENTAL PROCEDURES

Investigation of Manufacturing Parameters

The usual production process for KDNBF starts with the oxidation of orthonitroaniline (o-NA) to benzofuroxan (BF). The nitration of BF yields 4, 6 - dinitrobenzofuroxan, (DNBF), which is reacted with potassium bicarbonate to yield KDNBF.



W. B. Hardy and R. A. Parent (Ref 3) suggested alternate processes, but these were rejected because of the expensive and not readily available raw materials required. Also, it was known that at least two major commercial manufacturers of KDNBF were using the usual process, and this fact provided another important reason for the decision to investigate the parameters of that process, especially since time and funding did not permit investigation of all combinations of conditions; but for each parameter studied at least two variants were investigated. Inauspicious,unpromising or duplicating alternates were eliminated to hold the number of alternates studied within reasonable limits.

Figure 1A shows the schematic flow chart of the route followed from o-NA to DNBF. Two grades of o-NA were treated, the first, a refined grade from Baker Chemical Co., the second, a practical grade from American Aniline Corp., obtained via Matheson Chemical Co.

The processes identified with Roman numerals in Figures 1A and B are listed below.

(Note: In some cases more than one batch of an intermediate was produced and larger batches were made.)

I. 20 g of o-NA was dissolved in 300 ml of methanol saturated with 50 g of potassium hydroxide. This solution was cooled to $0^{\circ}C$, and 222 ml of 5.25 percent sodium hypochlorite (household bleach) was slowly added. The temperature of the reaction was held below $10^{\circ}C$. The BF precipitate was filtered and washed once with ice water. The yield was 95 percent of BF with a melting point of $66-67.5^{\circ}C$.

II. 20 g of o-NA was dissolved in 300 ml of 83-percent methanol at $40^{\circ}C$. A mixture of 225 ml of household bleach and a solution consisting of 24 g of sodium hydroxide in 50 ml of water was added while the reaction temperature was held at $40^{\circ}C$ or less. The BF was precipitated by cooling to $0^{\circ}C$, filtered, and washed four times with ice water. The yield was 95 percent of BF with a melting point of $67-67.5^{\circ}C$.

III. Approximately 20 g of BF was recrystallized from 100 ml of methanol, filtered, and dried without washing. The yield, upon cooling to $50^{\circ}C$, was approximately 80 percent of BF with a melting point of $67-68^{\circ}C$.

IV. 26 g of BF was slowly dissolved in 260 g of concentrated sulfuric acid so that the temperature did not exceed 10°C. To this solution was added very slowly (70 minutes) a cold mixture of 130 g of concentrated sulfuric acid and 130 g of 90 percent nitric acid, which had been premixed at 10°C or below. The nitration was carried out below 20°C. After all the mixed acid had been added, the reaction mixture was momentarily heated to 45°C and then immediately quenched by pouring on a 2-liter volume of ice cubes. The DNBF precipitate was washed three times with 400 ml of ice water on the filter and dried. This process yielded 70 percent of DNBF with a melting point of 164-169°C.

V. This process was similar to Process IV, except that the cold mixture was prepared by adding only one part (by weight) of nitric acid (98 percent) to three parts of the concentrated sulfuric acid.

VI. This process was nearly identical with Process IV, except that the reaction mixture was held at 50°C for three minutes before pouring on ice.
(Note: The DNBF from V and VI subsequently was not used.)

VII. Approximately 200 g of KDNBF (P. A. Lot 7172) was treated with an excess of 50 percent acetic acid and digested on a steam bath for several hours. The crude DNBF so obtained was dissolved in glacial acetic acid and crystallized by cooling the solution to 5°C for 72 hours, and subsequently washed with water and recrystallized four times from the glacial acetic acid. The hot mother liquor was treated each time with activated carbon. The yield was approximately 10 percent of DNBF with a melting point of 172.8 - 173°C.

VIII. 50 g of DNBF (Process IV) was dissolved in 350 ml of glacial acetic acid to form a saturated solution at 95°C. A yield of 67 percent of DNBF (melting point 172-173°C) was obtained upon cooling the solution to 20°C, filtering the product and washing it four times with water.
(Note: Dilution of the mother liquor with water resulted in recovery of virtually all the DNBF in the liquor. The material so recovered was similar in appearance and melting point (166.5-170°C) to the original DNBF from Process IV.)

Three grades of DNBF were selected for processing to KDNBF as indicated in Figure 1B by the designations A (refined, B (crude),

and C (purest). These three grades were treated as shown in that figure. The processes are listed below

IX. 17 g of refined DNBF was dissolved in 1500 ml of 1/1 (by volume) acetone/water at 45°C. 7.5 g of potassium bicarbonate dissolved in 75 ml of water was added. The resulting solution was cooled to -10°C. The solid product was filtered and washed twice with cold acetone. A yield of 7.5 g of KDNBF in the form of fine flake crystals was obtained.

X. 28.5 g of crude DNBF was suspended in 750 ml of 1/4 (by volume) methanol/water at 40°C. 12.5 g of potassium bicarbonate dissolved in 50 ml of water was added. The solid product was filtered and washed four times with ice water and twice with methanol. The yield was approximately 90 percent of fine, gummy, crude KDNBF that dried to a hard caked mass.

XI. 5.65 g of the purest DNBF was dissolved in 500 ml of 1/1 (by volume) acetone/water at 45°C. 1.8 g of potassium carbonate dissolved in 50 ml of water was added without further heating. The mixture was stirred for 15 minutes while it was cooling naturally from 40 to 35°C. Then it was further cooled in an ice-salt bath to 0°C (twenty minutes) and held at 0°C for ten minutes. The product was washed twice by decantation with cold 1/1 (by volume) acetone/water, filtered, and dried. The product obtained, 70 percent of KDNBF in the form of large equant crystals, is shown in Figure A3 (see Appendix A). These crystals are designated automatic-loading grade, Type I.

XII. The fine flaky crystals obtained in Process IX were recrystallized from water by dissolving 7.5 g of KDNBF in 750 ml of water containing 1.5 g of potassium bicarbonate at 75°C, and cooling the solution to 5°C. The 6.5 g of solid product (KDNFB) obtained was filtered, washed twice with ice water and twice with methanol, and dried. These fine equant crystals are shown in Figure A4 (see Appendix A). They are designated automatic-loading grade, Type II.

XIII. Approximately 3 g of crude KDNBF in the form of wet cake (Process X) was dissolved at 80°C in 300 ml of water made slightly alkaline (pH 8.5) by addition of 0.1 g of potassium bicarbonate. After cooling the solution to 0°C, a 50 percent yield of KDNFB in the form of a flaky crystalline product similar to the commercial hand-loading grade shown in Figure A5 (Appendix A) was obtained.

Evaluation of Various KDNBF's

Samples of KDNBF from three commercial sources were procured for evaluation and comparison with the various grades of KDNBF produced according to the procedures given above. Since revelation of the particular properties of these commercial KDNBF's could be in violation of proprietary rights, no identification of the companies involved is made in the report.

The evaluation tests applied to the various KDNBF's can be divided into two groups. The first group concerns the physical and chemical properties of KDNBF as follows:

1. Melting point (raw materials only)
2. Elemental analysis
3. Differential thermal analysis
4. Ultraviolet and infrared spectroscopy
5. Nuclear magnetic resonance
6. X-ray diffraction
7. Contamination by methanol-soluble sustances

The second group concerns the properties of KDNBF associated with its use in military items:

8. Vacuum stability test
9. Picatinny Arsenal Explosion Temperature Test
10. Picatinny Arsenal Impact Sensitivity Test
11. Bulk density
12. Sieve analysis
13. Pressure-time relationship in a 1-ml bomb

Most of these tests are fully described in the Draft Purchase Description, Appendix A. Tests not included in that appendix are described below:

Melting point determinations were conducted on a Thomas-Hoover capillary melting point apparatus with a heating rate of 1/2 to 1 degree centigrade per minute except as noted.

The ultraviolet spectroscopy was performed in methanol solution on a Beckman DK-1 recording spectrophotometer. The infrared spectroscopy was carried out using 0.5 percent KDNBF in potassium bromide pellets on a Perkins-Elmer Model 21 recording infrared spectrophotometer.

The Picatinny Arsenal Explosion Temperature Test is described in MIL-STD-650 (Ref 5), and the Picatinny Arsenal Impact Sensitivity Test in AMCP 706-177 (Ref 6).

REFERENCES

1. N. E. Brown, M. A. Cook and F. A. Olsen, The Structure and Isothermal Decomposition Kinetics of Salts of 4, 6-dinitrobenzofuroxan, Department of Metallurgy, University of Utah Technical Report PO 13-7664, July 22, 1965

2. A. Anzalone, J. Abel and A. Forsyth, Characteristics of Explosive Substances, Picatinny Arsenal Technical Report 2179, May 1955

3. W. B. Hardy and R. A. Parent, U.S. Patent 3, 163,561

4. Memorandum from T. S. Costain to Chief, NIKE-X Engineering Division; response to WO 4366-64 calling for a study of KDNBF/Binder Systems, 5 August 1969

5. MIL-STD-650, Explosive: Sampling, Inspection, and Testing

6. AMCP 706-177, U. S. Army Materiel Command, Washington, D. C. Properties of Explosives of Military Interest, March 1967

TABLE 1

Melting point determinations

Material	Melting Point Range, °C	Remarks
DNBF-42	171-173	5X recrystallized; heated 4°C/min
DNBF-42	171.5-173.0	5X recrystallized; heated 4°C/min
DNBF-42	172.8-173.0	Slower heating, 0.5°C/min
BF-46	67-68	Methods I and III (Fig 1) 4°C/min
BF-46	67.7-68.2	Slower heating, 0.5°C/min
BF-47	67.0-67.5	Method II (Fig 1) 0.5°C/min
BF-48	67.5-68.0	Repeat batch Method II 0.5°C/min
BF-58C	66.0-67.5	Method I (Fig 1) 0.5°C/min
BF-58R	67.2-68.2	Recrystallized BF-58, 0.5°C/min
DNBF-60A	160-165.5	Method VI (Fig 1) from BF-47, 1°C/min
DNBF-60B	166.5-170	Method IV (Fig 1) from BF-47, 1°C/min
DNBF-60C	154.5-160	Method V (Fig 1) from BF-46, 1°C/min
DNBF - 60D	163-168	Method IV (Fig 1) from BF-58R, 1°C/min
DNBF-60E	164-169	Method IV (Fig 1) from BF-58C, 1°C/min
DNBF-64A	172-173	Method VIII (Fig 1) from DNBF-60D and -60E
0-NA	70.8-71.0	Baker lot No. 1-3581, MP given - 71-72°C
0-NA	69.4-70.2	Matheson's practical grade, no MP given
Vanillin	80.7-81.2	0.5°C/min, literature: 81-82°C
Sulfanilamide	163.5-165.0	0.5°C/min, literature: 164.5-166.0°C
Sulfapyridine	191.0-192.0	0.5°C/min, literature: 190-193°C

TABLE 2

Elemental analysis

KDNBF

Sample Number	Identification	Percent C	H	N	K
45	Auto-loading grade, Type I (purest)	25.95	1.26	18.71	14.39
52A	Repeat of 45 under new number	26.02	1.17	18.91	13.73
52A	Repeat of 45 under new number	26.09	1.27	18.73	13.90
66B	Auto-loading grade Type II	26.07	1.11	23.31	13.10
66B	Auto-loading grade Type II	26.28	.86	22.94	12.76
67R	Hand-loading grade similar to commercial	26.38	0.91	24.87	13.41
67R	Hand-loading grade similar to commercial	26.07	0.90	25.43	13.47
X	Commercial from company X	26.47	0.78	22.65	14.05
X	Commercial from company X	26.59	1.05	22.25	13.80
Y	Commercial from company Y	25.65	0.71	22.63	13.43
Y	Commercial from company Y	25.95	0.68	23.03	13.51
Z	Commercial from company Z	26.08	0.74	23.56	13.13
Z	Commercial from company Z	26.04	0.99	22.90	13.33
78	Crude, unacceptable	26.64	0.92	26.16	12.73
78	Crude, unacceptable	26.42	1.11	25.81	12.73
310	Lot SMUPA 7172, made at PA in 1965	24.96	1.41	18.33	13.33
310	Lot SMUPA 7172, made at PA in 1965	25.40	1.26		
52B	Lot SMUPA 7172, resubmitted, new No.	25.48	1.21	18.57	14.67
52B	Lot SMUPA 7172, resubmitted, new No.	25.40	.96	18.90	15.02
52B	Lot SMUPA 7172, resubmitted, new No.	25.67	1.32	18.25	14.61

TABLE 2 (cont'd)

Sample Number	Identification	Percent C	H	N	K
	Decomposition Products				
31SLR	Soluble material recovered from super-natant alcohol over KDNBF	37.24	2.80	14.80	10.92
	SMUPA Lot 7172, after 5 years storage	37.44	2.94		
52C	Acetone-soluble fraction (90%)of product	29.08	1.31	20.22	10.74
	resulting from boiling KDNBF six hours	29.32	1.51	20.23	10.03
	in large excess of water	29.07	1.49	20.05	10.43
31T	SMUPA 7172 boiled in Toluene	25.09	1.29	19.31	12.63
31T	SMUPA 7172 boiled in Toluene	25.20	1.22		
31TR	31T recrystallized from water	25.30	1.64	18.13	13.21
31TR	31T recrystallized from water	25.34	1.63		
None	Theoretical composition $C_6H_3N_4O_7K$	25.54	1.07	19.85	13.86
	DNBF				
60A	Washed nitration product (MP160-165.5)	31.68	1.17	24.56	42.49
60A	Washed nitration product (MP 160-165.5)	31.59	1.02	24.46	42.17
60E	Washed nitration product (MP 164-169)	32.21	0.67	24.65	41.10
60E	Washed nitration Product (MP 164-169)	31.95	0.76	24.80	41.22
57B	Washed nitration product (MP 166.5-170)	31.78	0.98	24.85	42.54
		31.76	0.72	24.92	42.76
42	Recrystallized 5X(MP 172.8-173.0)	32.07	0.94	24.66	42.00
None	Theoretical composition $C_6H_2N_4O_6$	31.9	.9	24.8	42.4

TABLE 3

Results of tests in the 1-milliliter pressure bomb

Sample No.	Identification	Sample Weight milligram	Time to Ignition milliseconds	Time to 90% Max Pressure, ms	Max Pressure psi/mg
1	KDNBF-Y	3.9	0.95	2.50	30.8
2	KDNBF-Y	3.3	1.08	2.50	49.7
3	KDNBF-Y	4.1	1.18	3.20	54.9
4	KDNBF-Y	2.8	1.09	4.00	42.1
5	KDNBF-Y	2.5	1.13	3.50	32.0
6	KDNBF-Y	2.8	1.08	1.13	28.6
7	KDNBF-Y	2.7	1.02	1.07	33.4
9	KDNBF-X	3.1	1.10	3.00	41.9
11	KDNBF-X	3.2	1.03	2.00	42.2
12	KDNBF-X	2.9	1.07	2.50	48.2
13	KDNBF-X	3.1	1.08	2.50	42.0
14	KDNBF-X	2.4	1.08	2.50	37.5
15	KDNBF-X	2.3	1.08	3.00	48.0
16	KDNBF-X	3.9	1.08	3.00	42.3
17	KDNBF-Z	1.8	1.10	4.00	34.4
18	KDNBF-Z	2.1	1.11	3.00	51.4
19	KDNBF-Z	2.7	1.07	2.80	40.8
20	KDNBF-Z	2.0	1.07	3.20	43.5
21	KDNBF-Z	2.7	1.11	3.00	49.0
22	KDNBF-Z	2.2	1.05	2.50	40.9
23	KDNBF-Z	2.8	1.02	3.30	50.7
25	KDNBF-45	2.7	1.10	1.80	44.5
26	KDNBF-45	2.7	1.12	2.00	47.4
27	KDNBF-45	2.2	1.12	2.15	59.0
29	KDNBF-45	2.6	1.12	1.33	38.5
30	KDNBF-45	2.9	1.10	1.70	60.4
31	KDNBF-45	3.3	1.30	1.80	54.5
32	KDNBF-45	2.6	1.10	3.00	44.2
33	KDNBF-45	3.4	1.40	2.40	62.4
34	KDNBF-45	2.2	1.08	2.50	69.0

TABLE 3 (cont'd)

Sample No.	Identification	Sample Weight, milligrams	Time to Ignition milliseconds	Time to 90% Max Pressure, ms	Max Pressure psi/mg
35	KDNBF-78	1.9	1.09	3.00	68.4
36	KDNBF-78	2.2	1.10	2.60	50.0
37	KDNBF-78	3.7	1.00	2.35	36.0
38	KDNBF-78	3.1	1.08	1.11	37.1
39	KDNBF-78	3.0	1.09	2.30	54.4
41	KDNBF-78	3.3	1.10	2.50	39.6
42	KDNBF-78	3.6	1.05	2.40	39.5
43	KDNBF-78	2.8	1.11	2.00	34.0
44	KDNBF-78	3.6	1.10	1.19	25.6

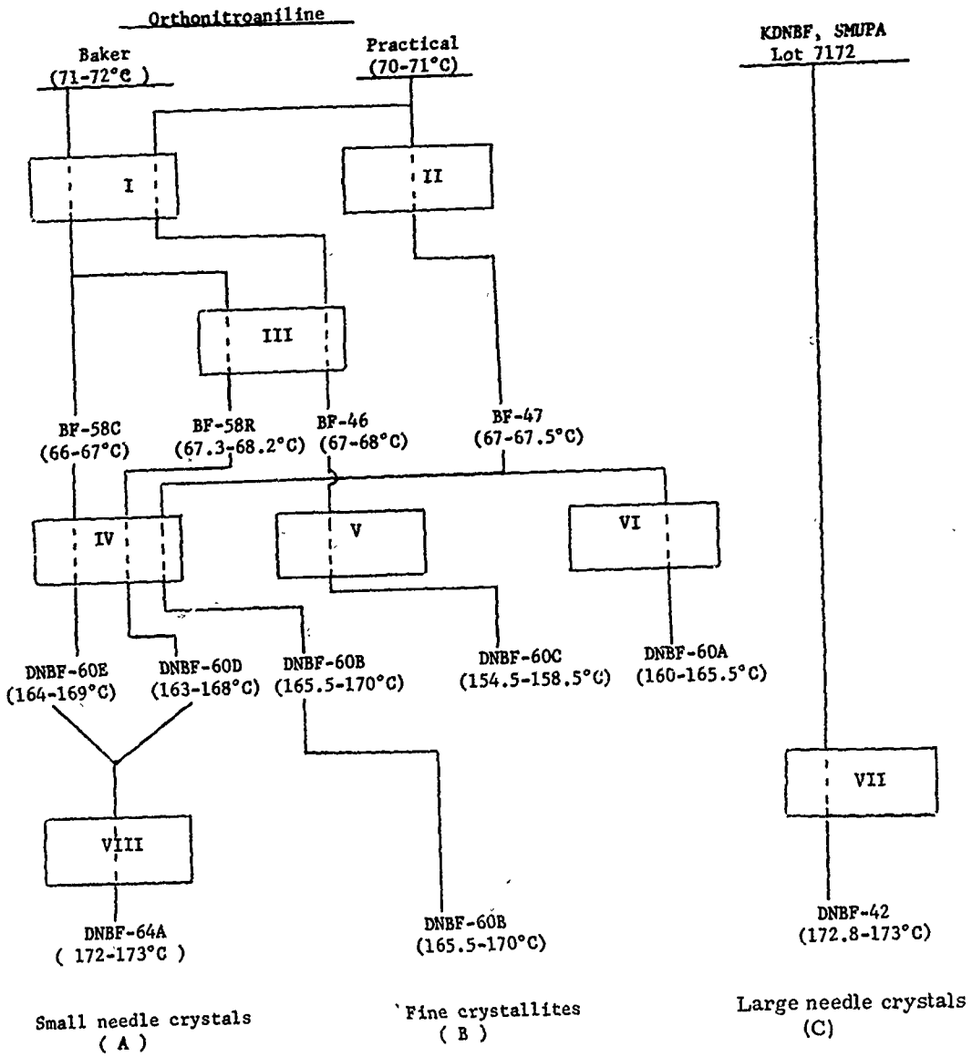


Fig 1A Flow chart

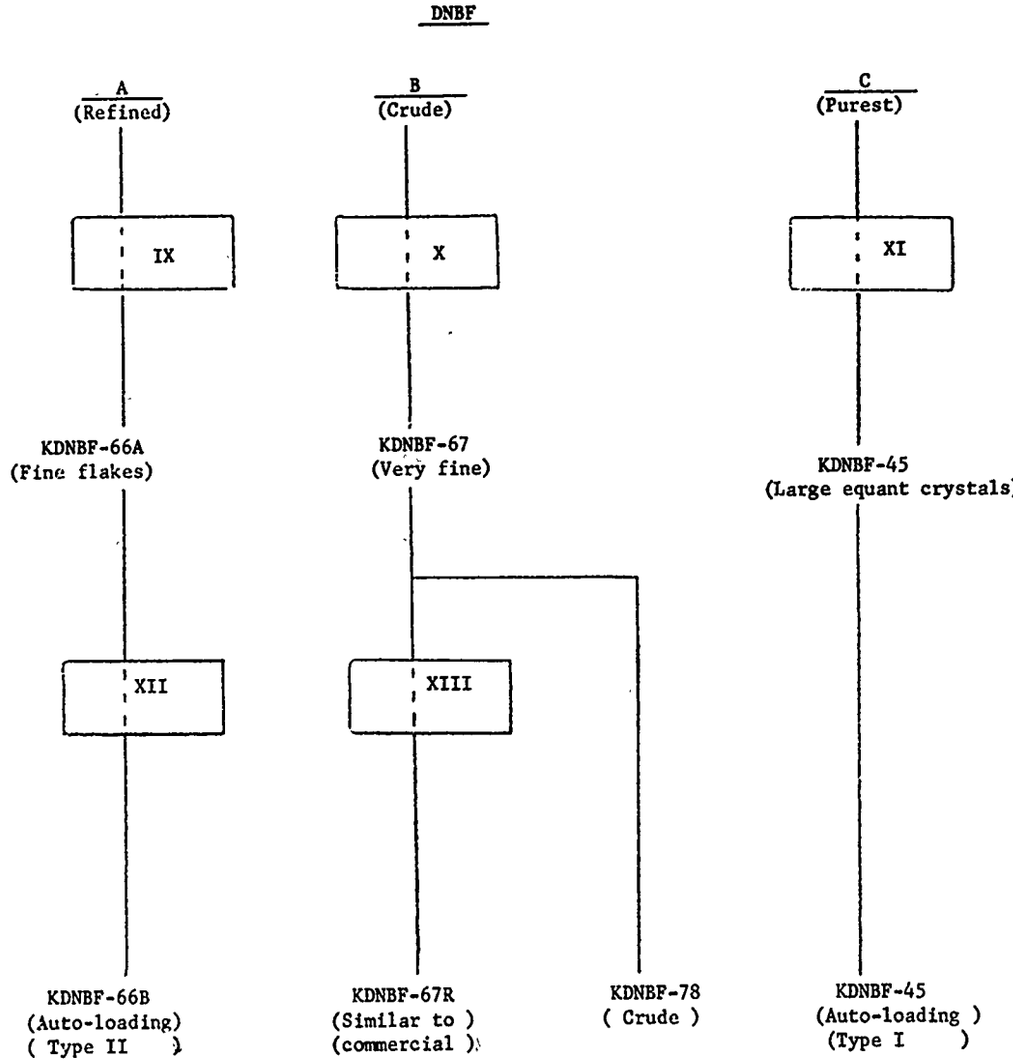


Fig 1B Flow chart

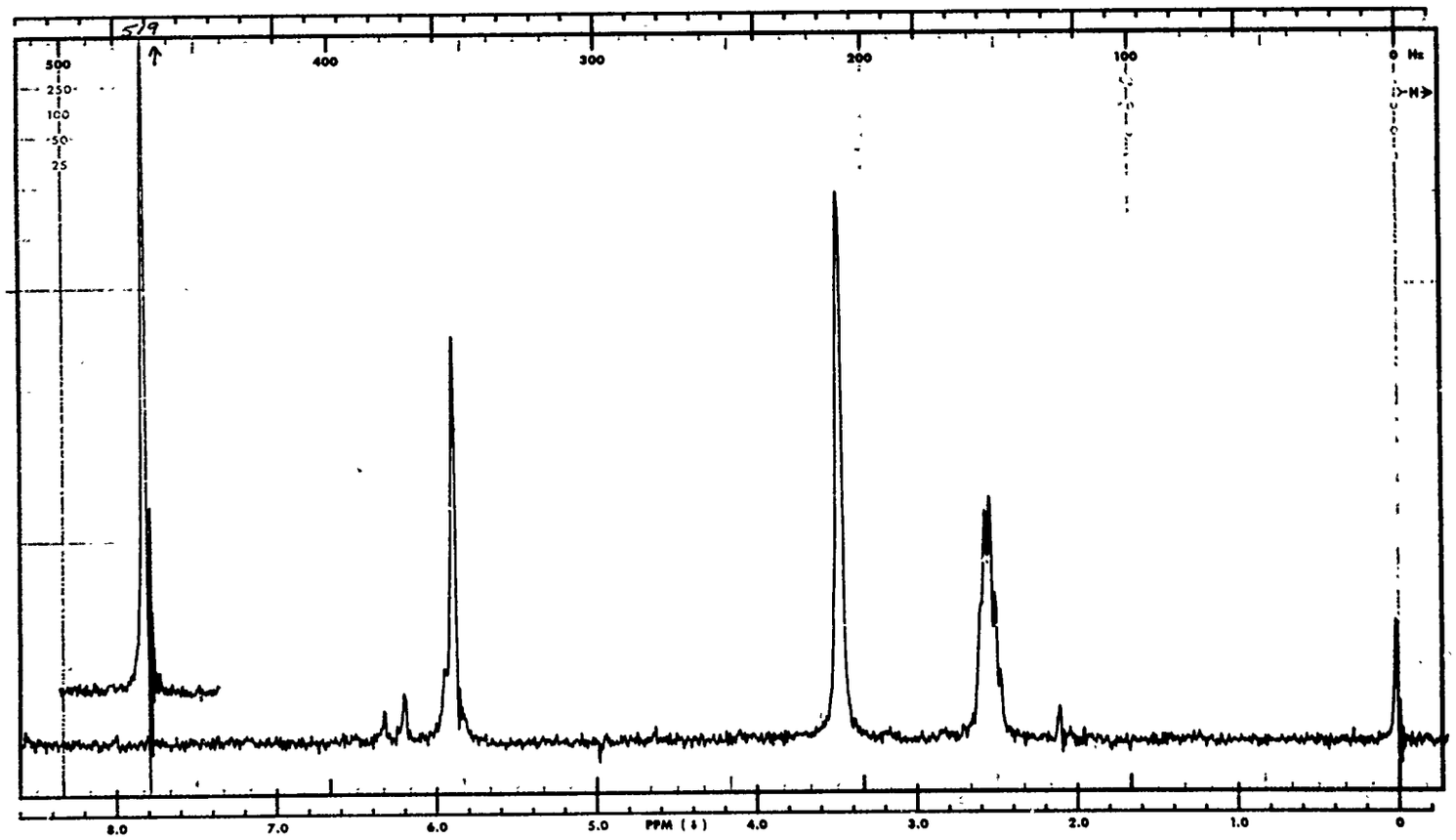


Fig 2 NMR for KDNBF-45 with D_2O added

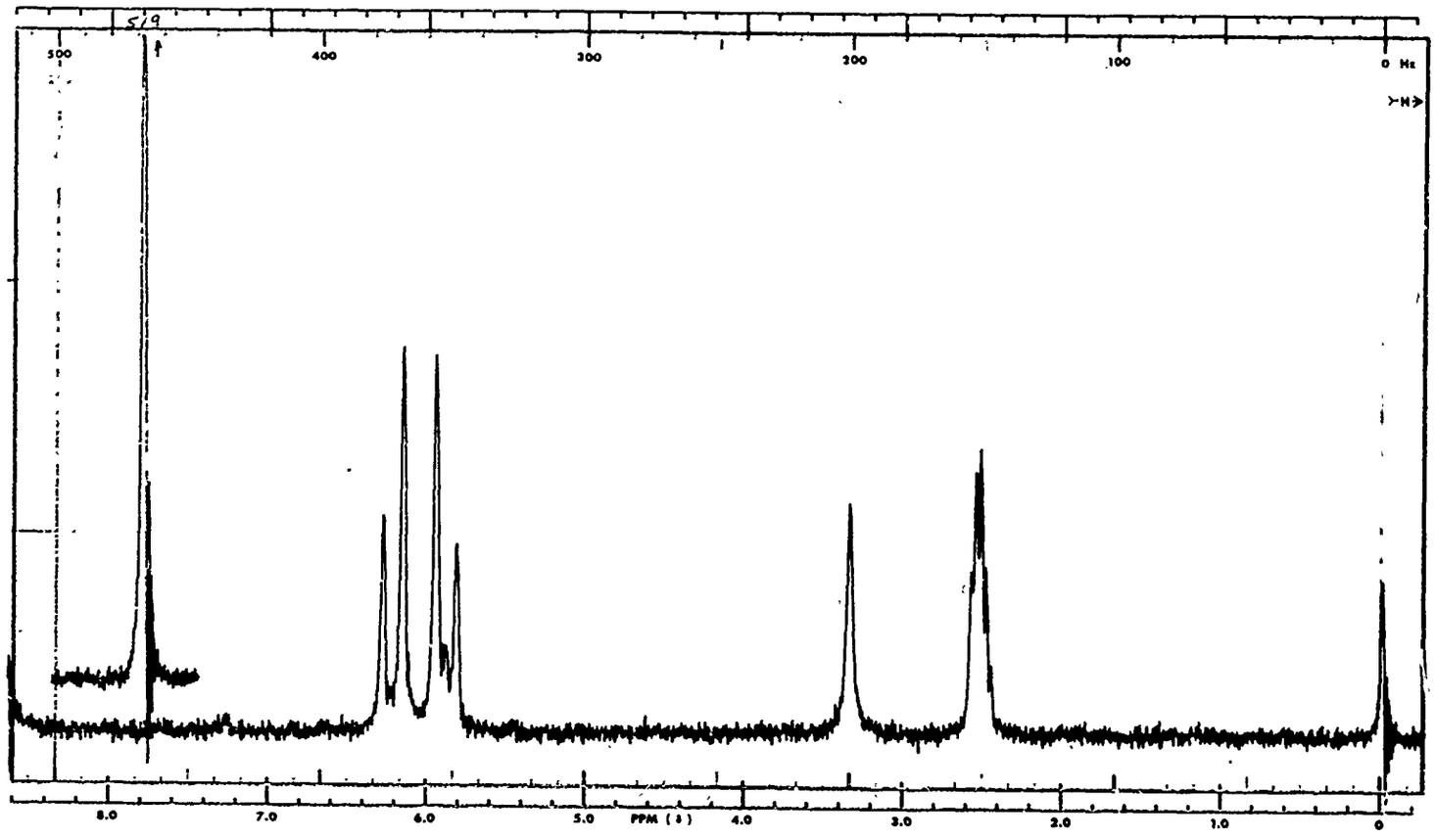


Fig 3 NMR for KDNBF-Y

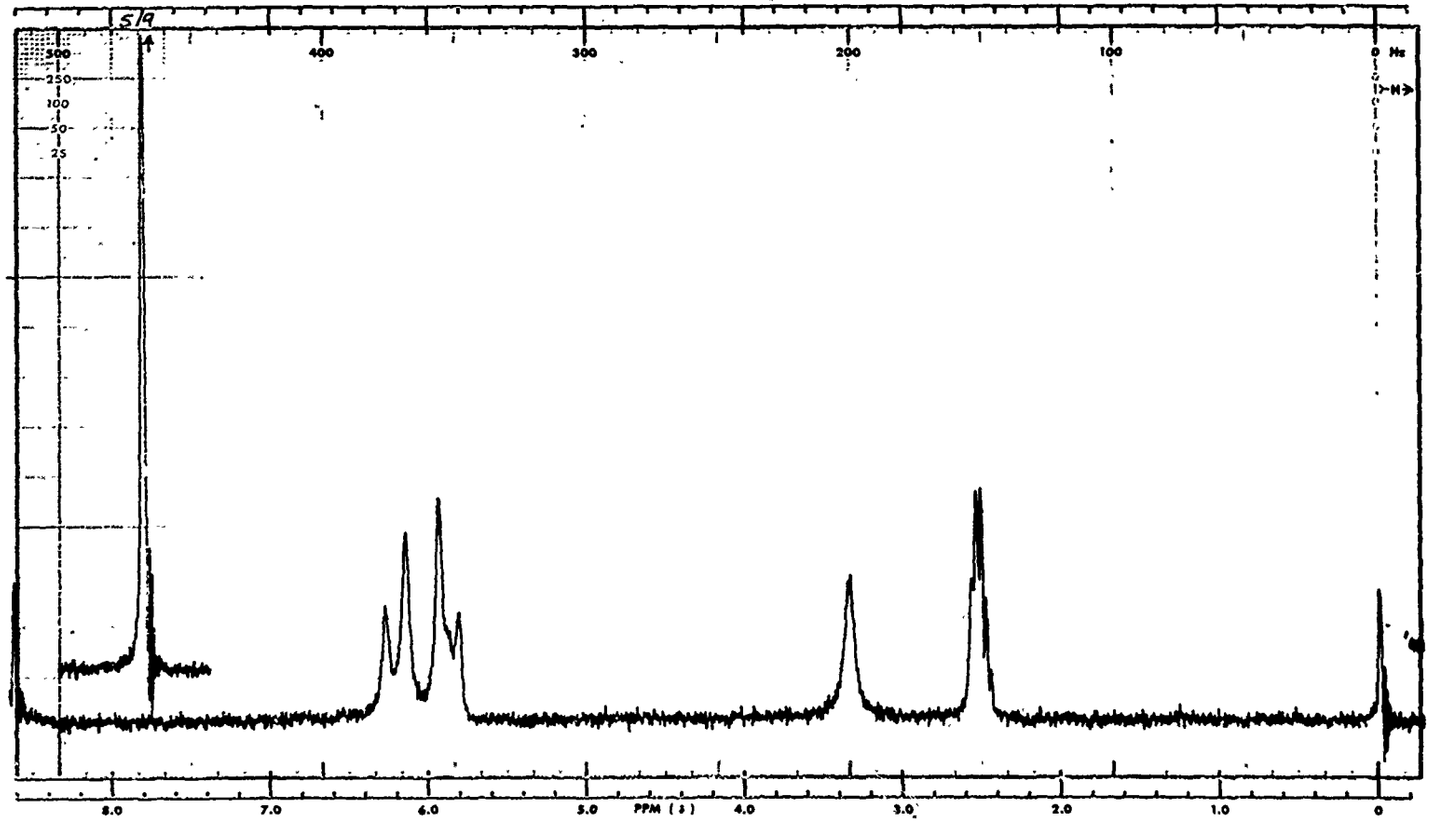


Fig 4 NMR for KDNBF-X

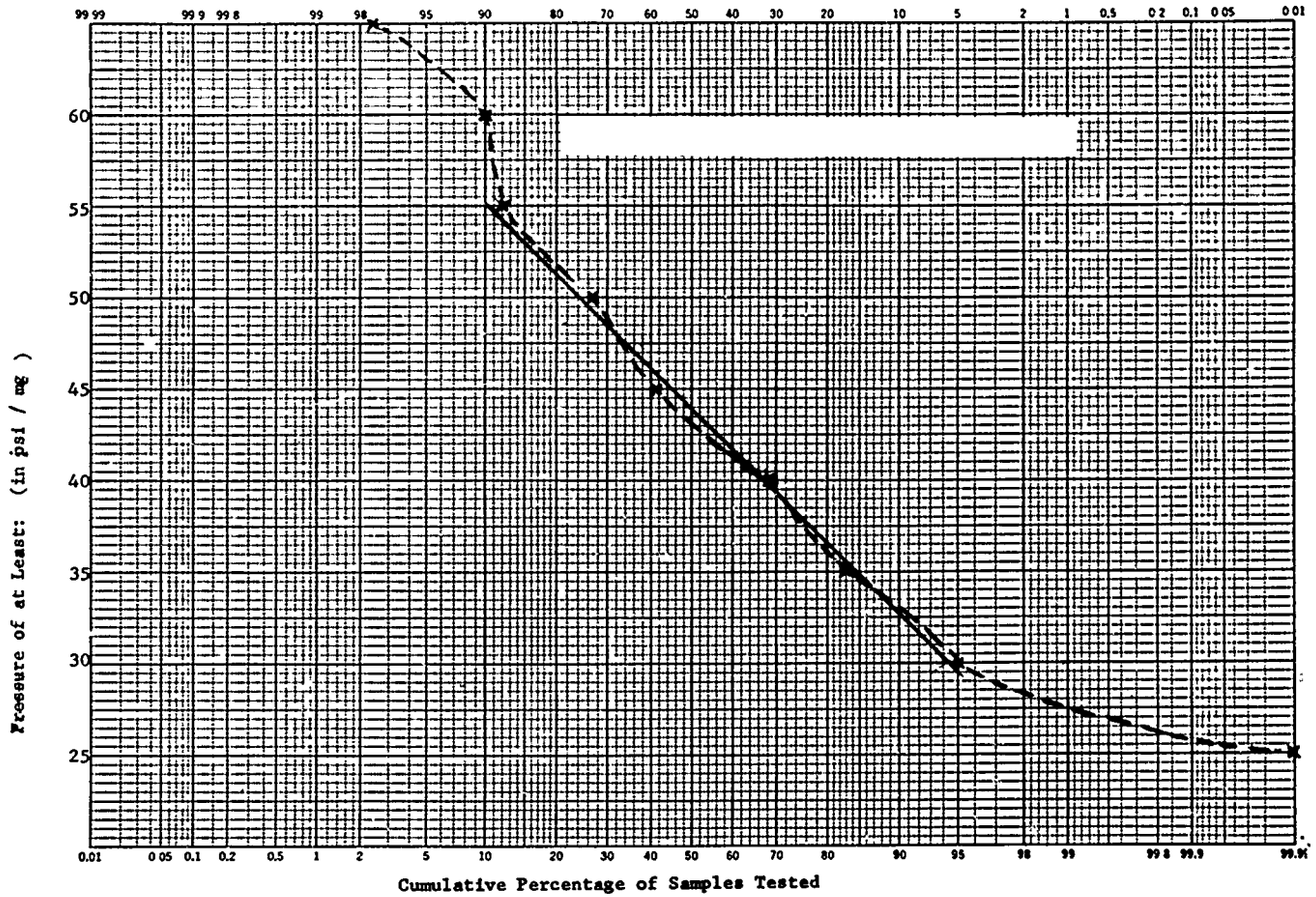


Fig 5 Pressures in 1-milliliter bomb

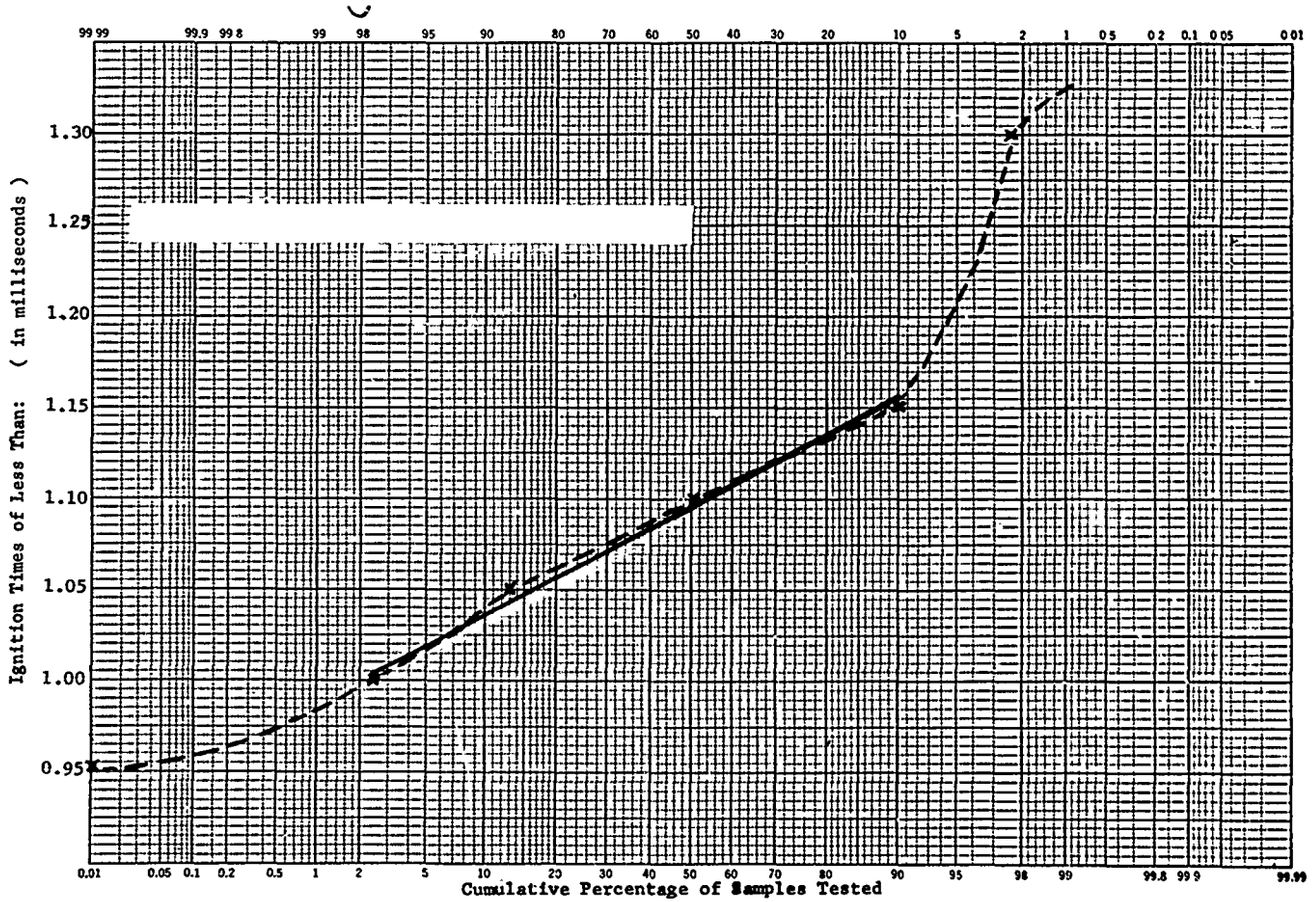


Fig 6 Ignition times in 1-milliliter bomb

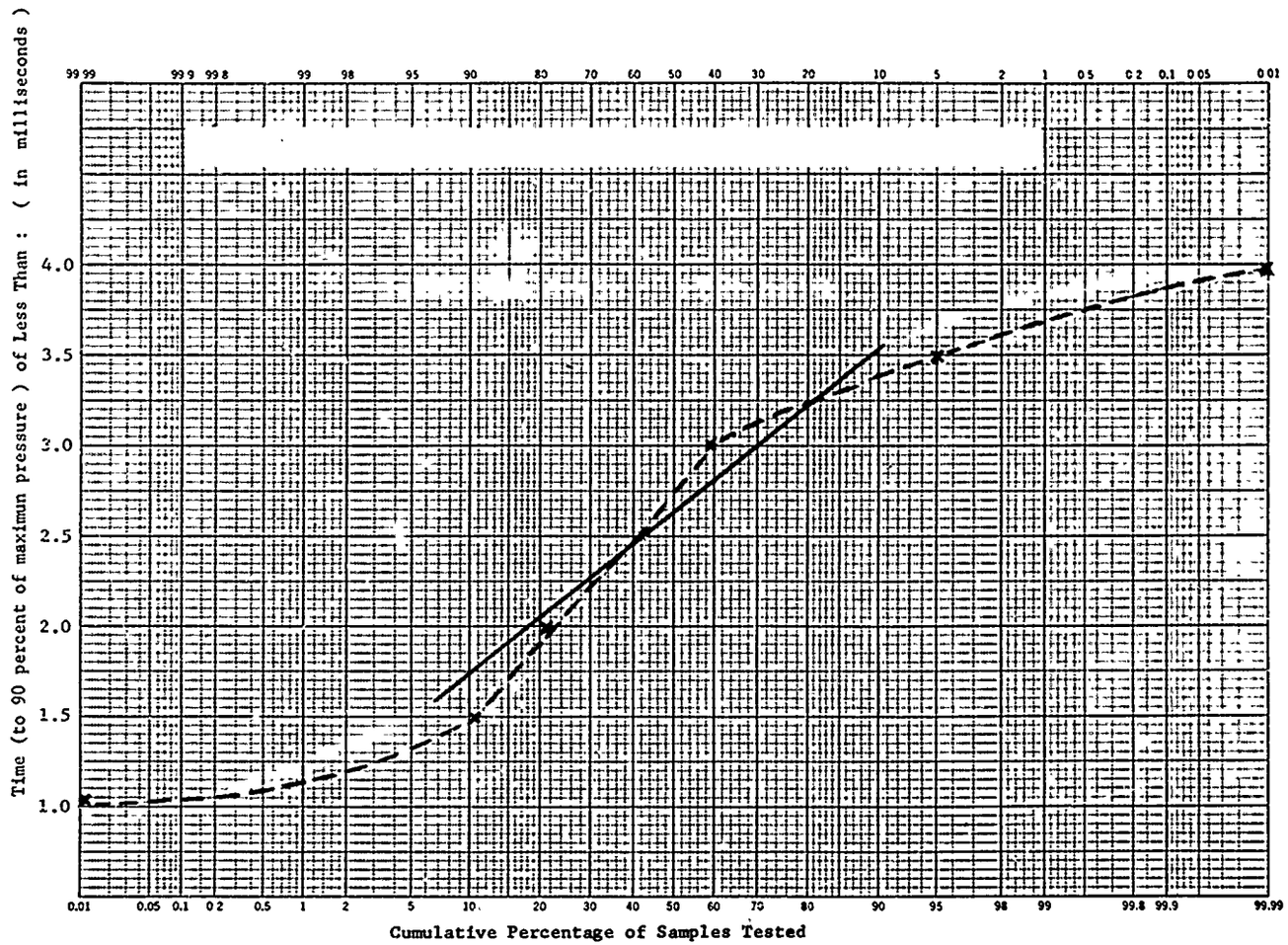


Fig 7 Time to 90 percent of maximum pressure in 1-milliliter bomb

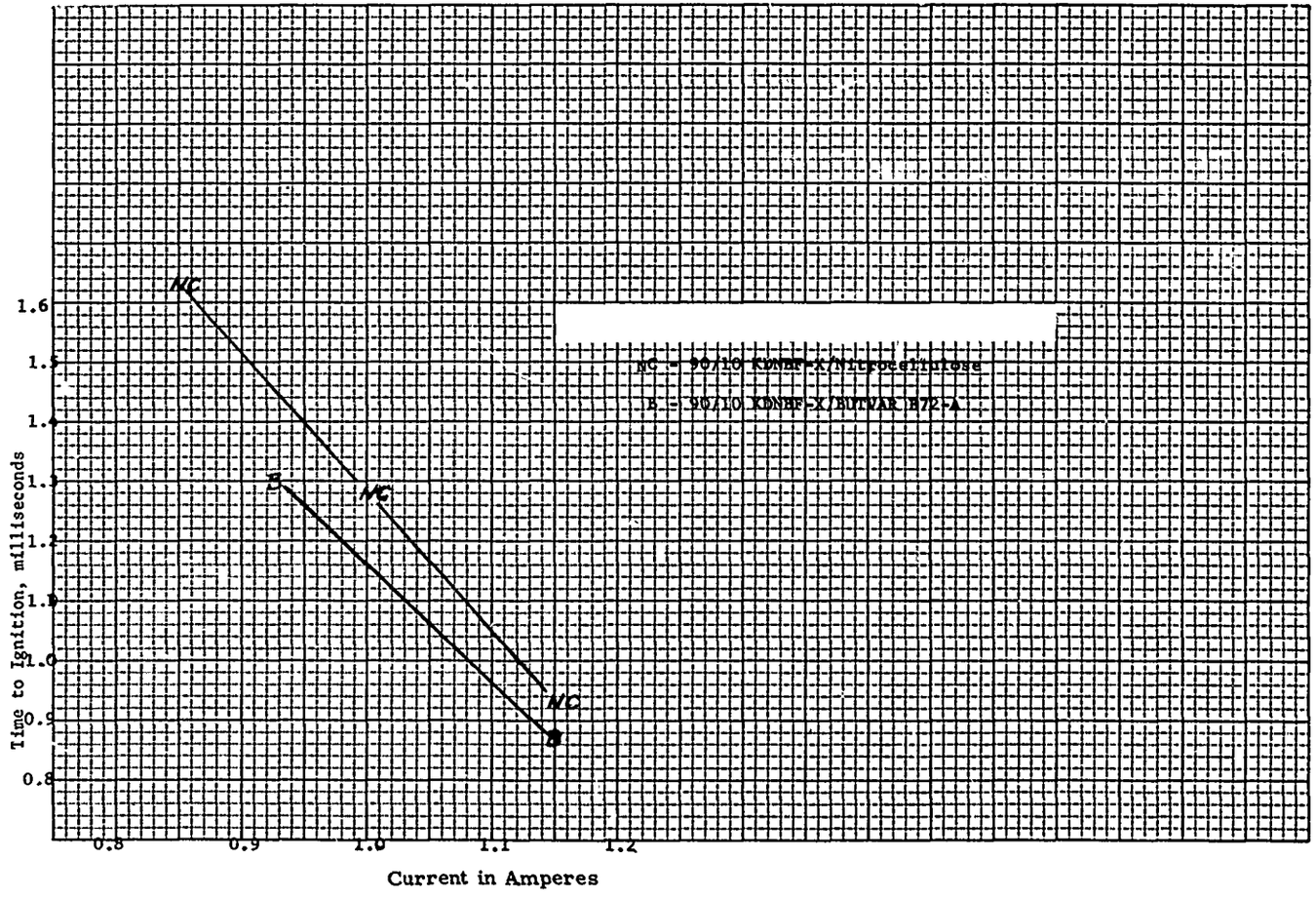


Fig 8 Time to ignition vs current

APPENDIX A

DRAFT PURCHASE DESCRIPTION

KDNBF
Potassium 4,6 - Dinitrobenzofuroxan

1. Scope

1.1 This draft purchase description covers the physical and chemical requirements of KDNBF for use in loading or as loaded into any explosive-actuated device. It does not include some quality assurance provisions and some packing and marking provisions, which are outside the purview of the Explosives Laboratory.

2. Applicable Documents

2.1 The following documents of the issue in effect on date of invitation for bids or request for proposals form a part of this purchase description to the extent specified herein.

Specifications
Military
(left blank)

Standards
Military
MIL-STD-650- Explosive: Sampling, Inspection, and Testing (Copies of specifications, standards, drawings, and publications required by supplier in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer.)

2.2 Other publications: The following document forms a part of this purchase description to the extent specified herein.

Code of Federal Regulations
49 CFR71-90 Interstate Commerce Commission
Rules and Regulations for the Transportation of
Explosives and Other Dangerous Articles

(The Interstate Commerce Commission Regulations are now a part of the Code of Federal Regulations (1949 Edition and Revisions) available from the Superintendent of Documents, Government Printing Office, Washington, D. C. Orders for the above publication should cite "49 CFR 71-90 (latest revision)".)

3. Requirements

3.1 KDNBF shall conform to the empirical formula $C_6H_3N_4O_7K$. It shall be manufactured according to the recommended method of manufacture (see para 7) or other methods which employ raw materials of no lesser purity than those specified in the recommended method.

3.2 Physical and chemical characteristics: KDNBF shall have the following chemical and physical characteristics when tested as specified herein:

3.2.1 Color: Red or Orange.

3.2.2 Form: Shall be determined by the intended use.

3.2.1.1 Automatic-loading grade: KDNBF intended for automatic loading operations (such as in Jones Machine) shall be in the form of equant crystals similar to those shown in Figures A3 and A4. The dry powder shall be easily dispersable and free from hard lumps or aggregates.

3.2.1.2 Hand-loading grade: KDNBF intended for hand operations may conform to 3.2.1.1, but shall at least be in the form of platelike crystals similar to those shown in Figures A5 and A6. The powder, when dried, may contain soft lumps and aggregates, but these must be easily dispersable by the process used to make the suspension-in-lacquer described in the fuctioning test described in 4.3.10.1.

3.2.3 Bulk density: 0.2 g/ml or greater.

3.2.4 Sieve analysis:

Auto-loading grade: Requirement to be established

Hand-loading grade: 90 percent passing through U. S. No. 50, but retained on U. S. No. 270. (Note: Record percentages on each sieve specified in 4.3.4. Percent lost shall be less than 5).

3.2.5 Differential Thermal Analysis (DTA): The graph of the DTA shall show one major exotherm between 210 and 225°C and be similar to those shown in Figures A-7, A-8, and A-9.

3.2.6 Nuclear Magnetic Resonance (NMR): The spectrum of the NMR shall exhibit at least the resolution, sharpness, and amplitude of the peaks as shown as examples of acceptable quality in Figures A-10 and A-11. Lack of resolution and amplitude as shown in Figure A-12 is cause for rejection.

3.2.7 X-ray Diffraction Pattern (XDP): The XDP shall conform to that illustrated in Figure A-13. The interplanar spacings shall conform to those given below in approximate order of intensity

3.02; 3.77; 5.24; 2.66; 2.18; 4.31; 3.98; 3.64; 3.45; 2.37; 6.46; 4.64; 3.29; 3.13

The XDP's for all types of KDNBF illustrated in this purchase description are the same.

3.2.8 Contamination by Methanol-Soluble substances: The solute content of the supernatant liquid in the storage container or containers shall not exceed 0.30 g per 100 ml or 0.5 percent of the KDNBF contained therein, whichever is less, when tested as specified in 4.3.7

3.2.9 Elemental Analysis: The following limits apply to the average of three determinations of elements contained in KDNBF when tested according to 4.3.9

C 25.4 - 26.4 percent

K 13.0 - 14.0 percent

3.2.10 Functioning Test: In at least 90 percent of the tests the KDNBF shall ignite (as indicated by commencement of pressure rise) within the limits of 1.0 - 1.2 milliseconds after flow of current through the bridgewire is started. In at least 90 percent of the tests, 90 percent of the maximum pressure rise shall occur within 3.5 milliseconds. Finally, in at least 66 percent of the tests the maximum pressure obtained shall be between 30 and 55 psi per mg of KDNBF per ml of bomb volume.

3.2.11 Vacuum Stability Test: One gram of KDNBF shall produce less than 1 ml of gas at 120°C when tested according to method 503.1 MIL-STD-650, dated 3 August 1962.

4. Quality Assurance Provisions

4.1 Lot Formation: A lot shall consist of one or more batches of KDNBF manufactured from one batch of dinitrobenzofuroxan (or equivalent precursor). A batch shall be defined as a quantity of material that has been subjected to the same unit chemical reaction process or recrystallization process intended to make the final product homogeneous. The physical mixing of parts of batches of different histories is in this context not considered as a process sufficient to qualify the resultant product as a homogeneous batch. It is the specific intent of this clause to prevent "working off" substandard batches by blending them with acceptable material.

4.2 Sampling: Each batch shall be sampled. If a batch is divided among several containers, a composite sample of equal portions from each container shall be taken.

4.2.1 Preparation of Samples: Visual inspection as described in 4.2.3.2 shall be performed on all containers holding a batch, and the additional testing prescribed in 4.2.3.2 shall be conducted, if necessary. All the supernatant absolute methyl alcohol under which the KDNBF has been stored shall be decanted into a filter fine enough to remove all suspended solids, leaving only the wetted solid in the container. These wetted solids shall be covered with twice their volume of absolute methanol and returned to storage. Alternatively, this decanting step can be combined with the normal drying procedure, in which case the original storage liquid must be collected separately from the wash liquids. The supernatant liquid shall be saved for the tests described in 4.3. The wash-liquid must also be saved. At least

5 g of sample per 100 g of KDNBF shall be taken from each container to form a total sample of at least 40 g. The 40-g sample shall be divided in half by quartering, one half being saved as a retention sample to be stored for five years, the other half to be used for the tests described in 4.3.

4.2.2 Visual Inspection: The supernatant liquid shall be light to dark-yellow in color. If one or more containers show a supernatant liquid of extremely dark color (burnt amber) so as to be almost opaque the container(s) shall be segregated and its (their) contents subjected to all tests specified for a batch as defined above. If the sample fails to comply with the requirements specified herein, the batch shall be rejected.

4.3 Test Methods and Procedures: The following tests shall be performed.

4.3.1 Color: The color of the KDNBF shall be determined by visual examination. For automatic loading (see 3.2.1.1) it usually is red to red-orange, for hand-loading orange with a metallic gleam due to reflections from the lamellar (thin flat) crystals. Color shall be recorded for information purposes only.

4.3.2 Form: This test is designed to judge the suitability of the KDNBF for either automatic or hand-loading. Material judged suitable for automatic loading will be acceptable for hand loading; but unless that material has been specifically procured for automatic loading operations, it need not conform to the standards specified for the latter.

4.3.2.1 Automatic-Loading Grade: On the end of a spatula a small (2 mg) sample of dry KDNBF shall be picked up and transferred to a clean glass microscope slide by gently tapping the spatula with the finger. The sample shall distribute itself over the glass slide as individual crystals with a minimum of loose aggregates. Subsequently, this material shall be examined under a microscope, using appropriate magnification between 100x and 300x according to crystal size. A clear high-contrast photomicrograph is to be retained for record and comparison. A material of equant (the three dimensions being nearly equal) crystalline shape similar to Figures A-3 or A-4 shall be submitted as a preproduction sample for determination of its suitability under actual loading conditions in the specific automatic loading equipment in which it is to used. If the preproduction sample is

judged suitable, it shall be used as a standard against which future batches shall be compared.

4.3.2.2 Hand-Loading Grade: Material conforming to 4.3.3.1 shall be considered suitable for use in hand loading. In addition, KDNBF in the form of lamellar crystals similar to those in Figure A-5 shall be acceptable. It probably will be found that the form of the crystals will have to be observed under the microscope with the KDNBF suspended as a thin slurry in water or alcohol. When such a slurry dries, the platelike crystals tend to agglomerate, but individual crystals may be observed at the edge of the main mass.

4.3.3 Bulk Density: The apparent bulk density shall be determined in conjunction with the sieve analysis (4.3.4). Approximately 2.0 grams of dry KDNBF will be weighed, accurately to .01 g. This material will be thoroughly slurried with 25 ml of absolute methanol contained in 25-ml graduated cylinder. The suspended KDNBF shall be allowed to settle for one hour, and the volume may be read at the line of separation between the KDNBF slurry and the relatively clear supernatant liquid.

4.3.4 Sieve Analysis: The KDNBF and methanol shall be reslurried in the 25-ml graduate. A stack of 3-inch-diameter plastic U. S U. S. Standard sieves numbers 50, 100, 200, and 270, which have been thoroughly cleaned, dried to constant weight at 75°C, and individually weighed to the nearest 0.001 g, shall be assembled, the joints between the sieves taped to make them reasonably airtight, and the stack set on a preweighed sheet of No. 1 filter paper (fast) in a close-fitting (about 90 mm) Buchner funnel, which in turn is held in a 500-ml vacuum flask. The assembly will be wetted with 25 ml of methanol, vacuum turned on at maximum flow, and the freshly suspended KDNBF slurry immediately poured into the sieve stack. Slight pressure on the sieve stack will maintain a seal so that the slurry is sucked through the screen stack within a few seconds. There may be a slight hangup if the No. 270 sieve contains over 50 percent of the total sample. Releasing the vacuum, swirling the sieves, and reapplying the vacuum will usually pull out the last of the methanol. The pulling of air through the sieve stack will be continued until the stack is virtually dry. The stack will be disassembled and dried to constant weight at 75°C, and each sieve and the sheet of filter paper weighed accurately to .001 gram. Finally, the percent of the original KDNBF sample retained on each sieve and on the filter paper will be

calculated and each percentage reported. Any difference between total weight recovered and original weight of sample shall be reported as percent lost.

4.3.5 Differential Thermal Analysis: The DTA shall be performed on a du Pont 900 Differential Thermal Analyzer at the following settings:

Reference: Glass beads

Rate: 10°C/minute

T: 50°C/inch

Δ T: 0.2°C/inch

Atmosphere: Helium at 100 ml/minute

Quantity: Approximately 0.5 mm of KDNBF shall be placed in the sample tube.

4.3.6 Nuclear Magnetic Resonance: Determine the NMR spectrum of KDNBF on a Varian Model T60 Spectrometer, or equivalent apparatus, under the following conditions:

Sweep Offset (Hz): 50	Sweep Width (Hz): 500
Spectrum Amplitude: 50.	Filter: 1 or 2
Spinning Rate (rps): 37-41	RF Power Level: .075
Sweep Time (sec): 250	Sample: 60 mg KDNBF in 0.4 ml DMSO-d_6

4.3.7 X-ray Diffraction Pattern: The sample shall be tested using Debye-Scherrer (powder method) technique with copper radiation applying a nickel filter.

4.3.8 Contamination by Methanol-Soluble Substances: The supernatant storage liquid collected in 4.2.3.1.1 shall be measured to determine the total volume to the nearest 10 ml per liter, and the weight of the KDNBF from which the supernatant liquid was removed shall be recorded for use in subsequent calculations. 10 ml of the supernatant liquid will be pipetted into each of three evaporating dishes, which have been dried to constant weight and weighed to the

nearest 0.0001 gram. The methanol will be allowed to evaporate at a temperature not to exceed 40°C. The dishes shall be dried to constant weight at 75°C and weighed to the nearest 0.0001 gram. The gains in weight will be recorded and averaged. The average gain will be multiplied by 10 and the result recorded as grams per 100 ml. The percentage solubility will be calculated based on KDNBF according to the following formula: percent =

$$\frac{\text{Total volume of supernatant liquid (ml) x grams of solute per 100 ml}}{\text{Total weight of KDNBF (grams)}}$$

4.3.8.1 Procedure if solubility exceeds 0.3 gram per 100 ml; The batch of KDNBF affected shall be reslurried with twice its apparent volume of absolute methanol, then over a period of three days remixed once a day to assure complete saturation of the supernatant liquid. If the KDNBF has been dried, the wash liquid shall be used as all or part of the methanol required. The test specified in 4.3.8 shall be repeated.

4.3.8.2 It should be noted that the purpose of this test is to detect high concentrations of a hydrolysis decomposition product which may be formed in KDNBF contaminated with water and stored for extended periods. The solubility, at room temperature, of the decomposition product is approximately 0.3 grams per 100 ml of methanol while that of KDNBF is approximately 0.03 gram per ml. Methanol saturated with decomposition product has an almost opaque burnt amber color.

4.3.9 Elemental Analysis: This analysis shall be conducted by standard methods of micro-chemical analysis.

4.3.9.1 Determination of Carbon: The analysis is conducted on a microcombustion train using about .001 gram sample mixed with V_2O_5. The sample is first heated to 220°C in a stream of oxygen. After the initial decomposition is finished, the temperature is increased to 1,000°C to complete the combustion. The analysis is completed by standard procedures.

4.3.9.2 Determination of Potassium: A .005 g sample of KDNBF will be dissolved in water. The potassium content will be determined by standard atomic absorption technique.

4.3.10 Functioning Test: The resin binder used in this test shall be Butvar B72A, a product of the Monsanto Chemical Corporation, St. Louis, Missouri. The lacquer used shall be made by dissolving 5 parts by weight (grams) of Butvar B72A in a solvent composed of 75 parts by volume (ml) of absolute ethanol and 25 parts by volume (ml) of toluene.

4.3.10.1 Bridgewire spotting mixture: A quantity of 0.250 ± .001 gram of KDNBF will be weighed out in a 10-ml teflon beaker. Exactly 0.556 ± .001 gram of 5-percent Butvar lacquer will be added. The mixture will be stirred with a thin wooden dowel (wooden applicator of stick-test type, for example) using a slow circular motion until a smooth lump-free suspension is formed. (A lumpy suspension is cause for rejection of the batch; see 3.2.1.2).

4.3.10.2 Ten plugs (Figure A-2) will be dried to constant weight at 75°C and individually weighed to the nearest .0001 gram. KDNBF/Butvar resin suspension will be applied to bridgewire of the plugs to form compact rounded spots, and air-dried for one hour, then dried to constant weight at 75°C. The spotted plugs will be reweighed to .0001 gram. The individual weights of the spots shall be between 2 and 4 mg (see Note 6.1).

4.3.10.3 The plugs shall be fired with a current between 0.95 and 1.1 amperes in a 1 ml ± 0.10 ml volume pressure bomb similar to that in Figure A-1. On a dual-beam oscilloscope record: the current in amperes, the time from start of current flow to commencement of pressure rise, time to 90 percent of maximum pressure, and the maximum pressure developed. Calculate psi/mg/ml by dividing maximum pressure developed by the particular weight of KDNBF spotting mixture on the bridgewire, and multiply the quotient by the determined volume of the bomb used.

4.3.11 Vacuum Stability Test: Duplicate tests are to be conducted at 120°C, using two 1-gram samples of KDNBF according to Method 503.1; MIL-STD-650, 3 August 1962.

5. Preparation for Delivery of Bulk KDNBF (see Note 6.2)

5.1 Preservation and Packaging: The reactivity of KDNBF with moisture necessitates a completely anhydrous storage condition. Only absolute methanol shall be used to wet the KDNBF. KDNBF shall

be placed in a methanol-impervious plastic container of such size that approximately twice its volume of absolute methanol can be added to nearly fill the container. Provision shall be made to insure complete wetting of the KDNBF with the absolute methanol. The containers shall be fitted with a nonscrew-thread closure which is liquid-tight. The closure shall be fastened with methanol-impervious nonmetallic tie, heat-shrunk film, or by other suitable methods to prevent accidental dislodgment of the closure.

5.2 Packing (to be completed by Plastics and Packaging Laboratory, FRL)

5.3 Marking (to be completed by cognizant Division)

6. Notes

6.1 Bridgewire plugs (Figure A-2) required for the above operation will be furnished by the contracting officer upon request.

6.2 KDNBF contained in end items shall be packed according to specifications covering the end items.

7. Recommended Manufacturing Procedure

recommended procedure follows the route ortho-nitroaniline (o-NA) to benzofuroxan (BF), benzofuroxan to 4, 6-dinitrobenzofuroxan (DNBF), and 4,6-dinitrobenzofuroxan to potassium dinitrobenzofuroxan (KDNBF). Great latitude is allowed for general procedural details such as batch size and solution concentration to allow individual contractors to adapt the process to their own needs. However, certain critical check points are defined where experience has shown close control is necessary, and the quality or grade of raw materials and intermediate compounds is specified.

7.1 Raw materials and intermediate compounds

a. Ortho-nitroaniline - MP 70°C minimum

b. Sodium or potassium hydroxide - ACS grade

c. Sodium hypochlorite - household bleach or equivalent

d. Methanol, absolute, ACS grade

e. Concentrated sulfuric acid - 95-98 percent ACS grade

f. Concentrated nitric acid - 90 percent minimum, ACS grade

g. Potassium bicarbonate - ACS grade

h. Potassium carbonate - ACS grade

i. Acetone - ACS grade

j. Benzofuroxan - MP 67°C minimum

7.2 Oxidation of o-NA to BF: The 0-NA is to be oxidized in methanol in the presence of strong concentrations of sodium or potassium hydroxide, using approximately 5 percent sodium hypochlorite solution. Reaction temperatures can be between 5 and 40°C, but shall not exceed the latter in order to prevent eutectic melting of BF and methanol. The resultant BF shall have a melting point range (capillary melting point apparatus) between 67°C minimum and 70°C maximum. BF may be recrystallized from methanol if necessary.

7.3 Nitration of BF to DNBF: BF dissolved in 10 parts of concentrated sulfuric acid shall be nitrated with mixed acid (i.e., nitric acid/sulfuric acid in a ratio of between 1:1 and 1:4) at temperatures between 0°C and 20°C. At the end of the addition of the nitrating acid, the mixture may be momentarily heated up to 50°C, but shall be immediately quenched on ice after attaining that temperature. The DNBF shall have a melting point range at some point between 166°C and 173°C. Typically, the washed product of nitration has an MP range of 166-170°C, determined in a capillary melting point apparatus. The DNBF may be recrystallized from glacial acetic acid or methanol, if necessary. Successive recrystallizations yield DNBF with an MP of 172-173°C.

7.4 Reaction of DNBF to KDNBF: DNBF is suspended or dissolved in water to which has been added from 20 to 50 percent of methanol or acetone. The reaction with potassium bicarbonate is carried out at a temperature of 40°C or below to prevent (as far as possible) hydrolysis of the product KDNBF. Unless conditions or ingredients

are closely controlled, the initial reaction yields a product which is difficult to wash and dries to a hard, caky mass. The crude KDNBF may be recrystallized from water to yield a product which may be acceptable for hand-loading grade (see 4.3.2.2). Some hydrolysis decomposition product will form during recrystallization, and the product will require sufficient washings with methanol to meet the requirements of 4.3.8.

7.4.1 Reactions to produce KDNBF of automatic-loading grade: The following reactions have been conducted on a laboratory scale and are presented only as a guide for extention to plant practice.

7.4.1.1 0.25 mole of DNBF (MP 172-173°C) in 500 ml of 50/50 water/acetone is reacted at 45°C with 0.13 mole of potassium carbonate dissolved in 50 ml of water. The mixture is stirred for 15 minutes while it cools to approximately 35°C, subsequently cooled in an ice-salt bath to reduce its temperature to 0°C within twenty minutes, and held for 10 minutes at 0°C. The product is washed once by decantation with 50/50 acetone and water chilled to 0°C, and once with the same wash on a vacuum filter. Air is sucked through the filter for 30 minutes or more to semidry the product. Drying is finished in an oven at 75°C. The product obtained is shown in Figure A-3.

7.4.1.2 7.5 grams of hand-loading grade KDNBF is dissolved in 750 ml of water containing 1.5 grams of potassium bicarbonate (pH-8.7) at 75°C. The solution is cooled within 10 minutes to 30°C and then in an ice-salt bath to 5°C within 30 minutes. The product is washed three times with cold methanol on a vacuum filter, air-dried and then oven-dried at 75°C. The product is shown in Figure A-4.

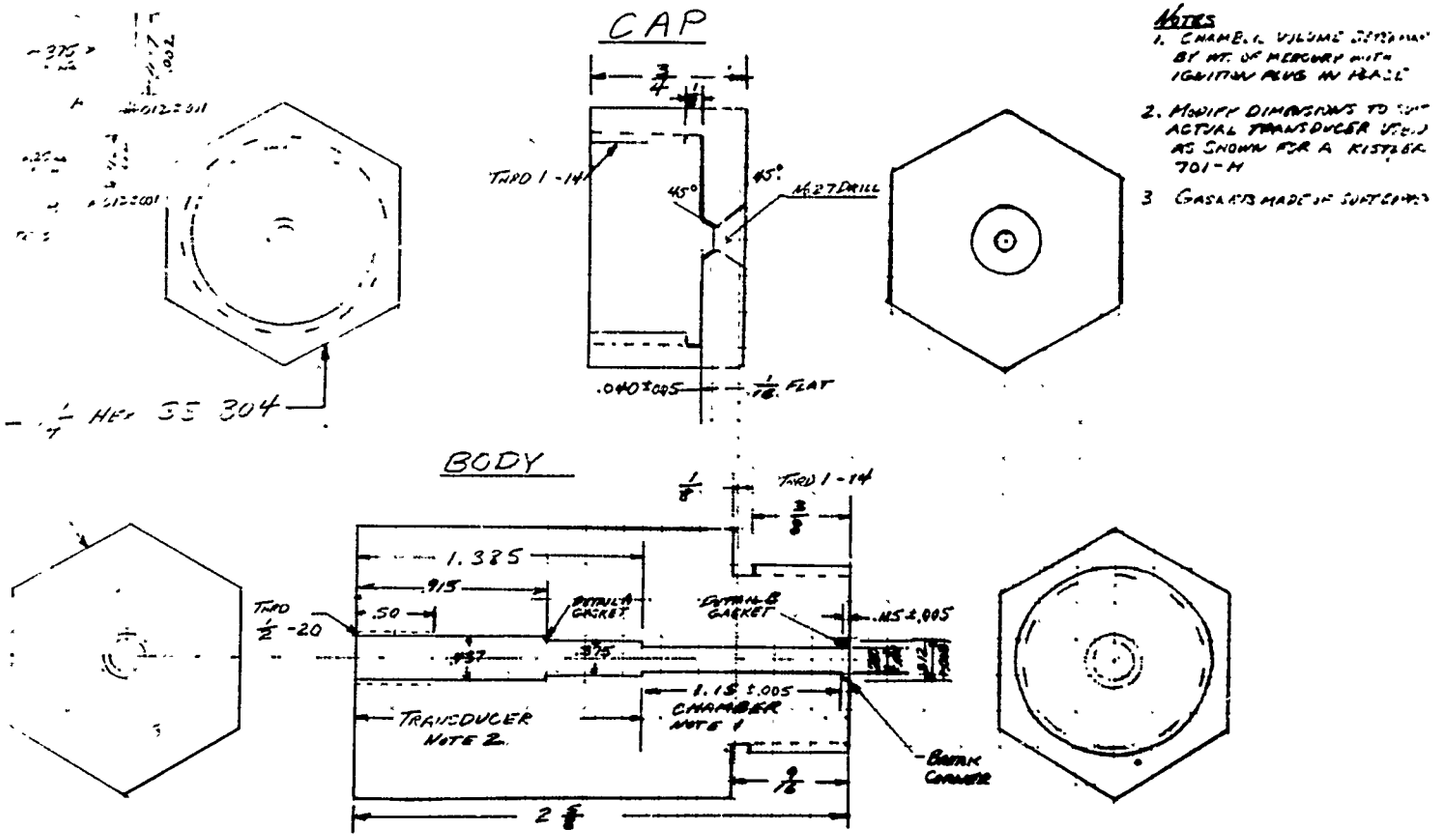
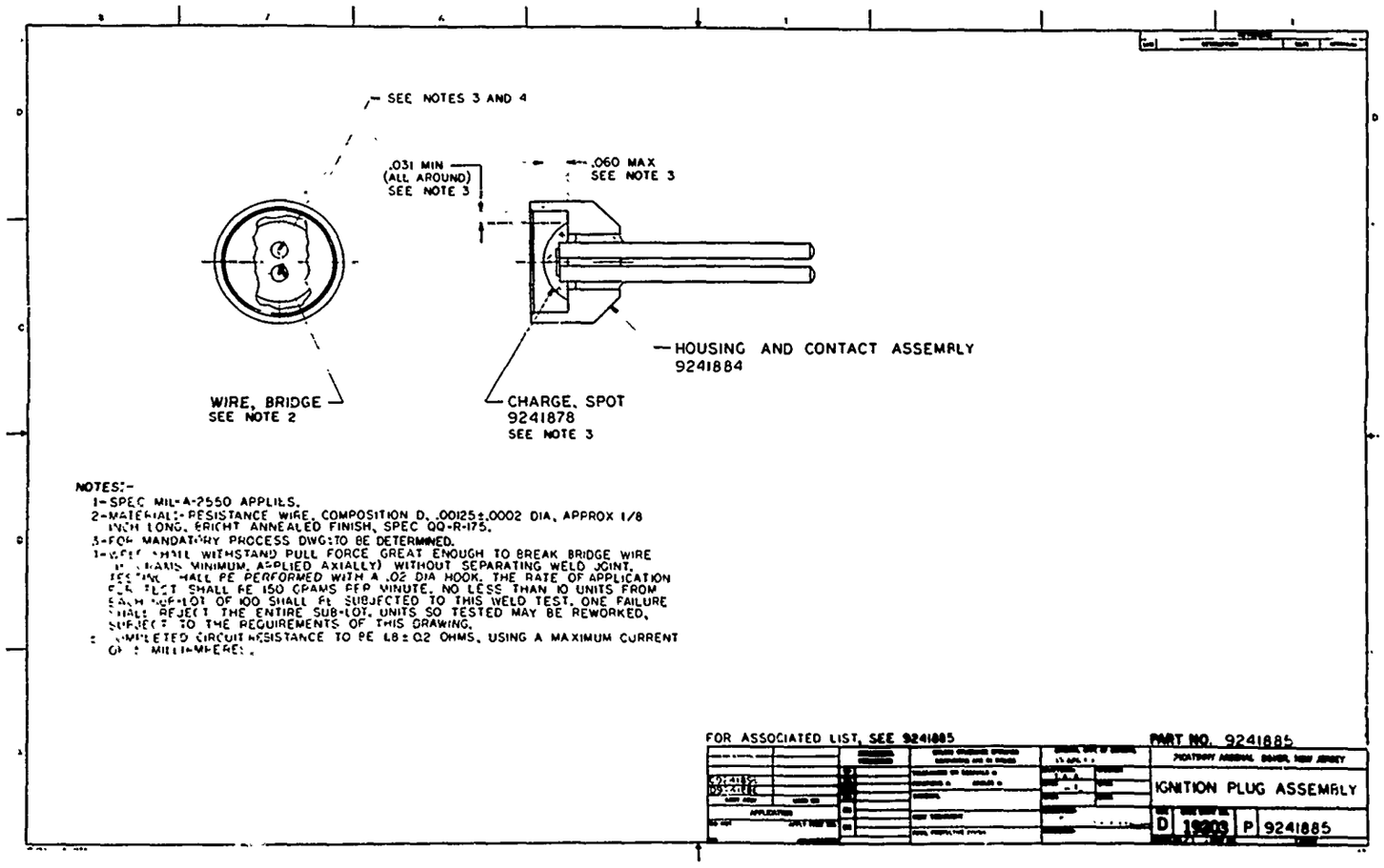


Fig A-1 Pressure bomb, 1 ml



NOT REPRODUCIBLE

Fig A-2 Ignition plug assembly

NOT REPRODUCIBLE

Fig A-3 KDNBF, auto-loading grade, Type I, 130x

Fig A-4 KDNBF, auto-loading grade, Type II, 130x

NOT REPRODUCIBLE

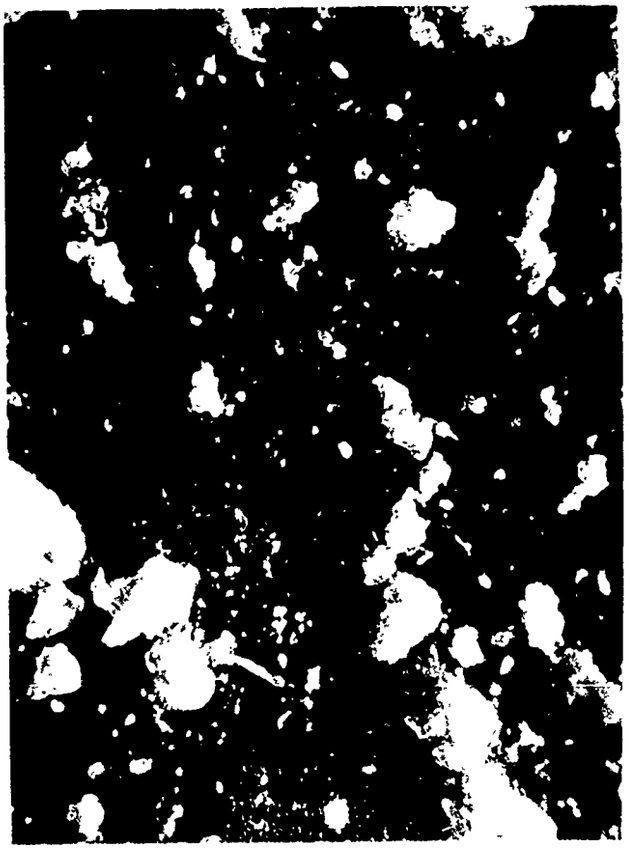


Fig A-5 KDNBF, hand loading grade, as ordinarily dried 130 x

NOT REPRODUCIBLE

Fig A-6 KDNBF, hand loading grade, thin slurry allowed to dry on slide. Edge of field 130 x

NOT REPRODUCIBLE

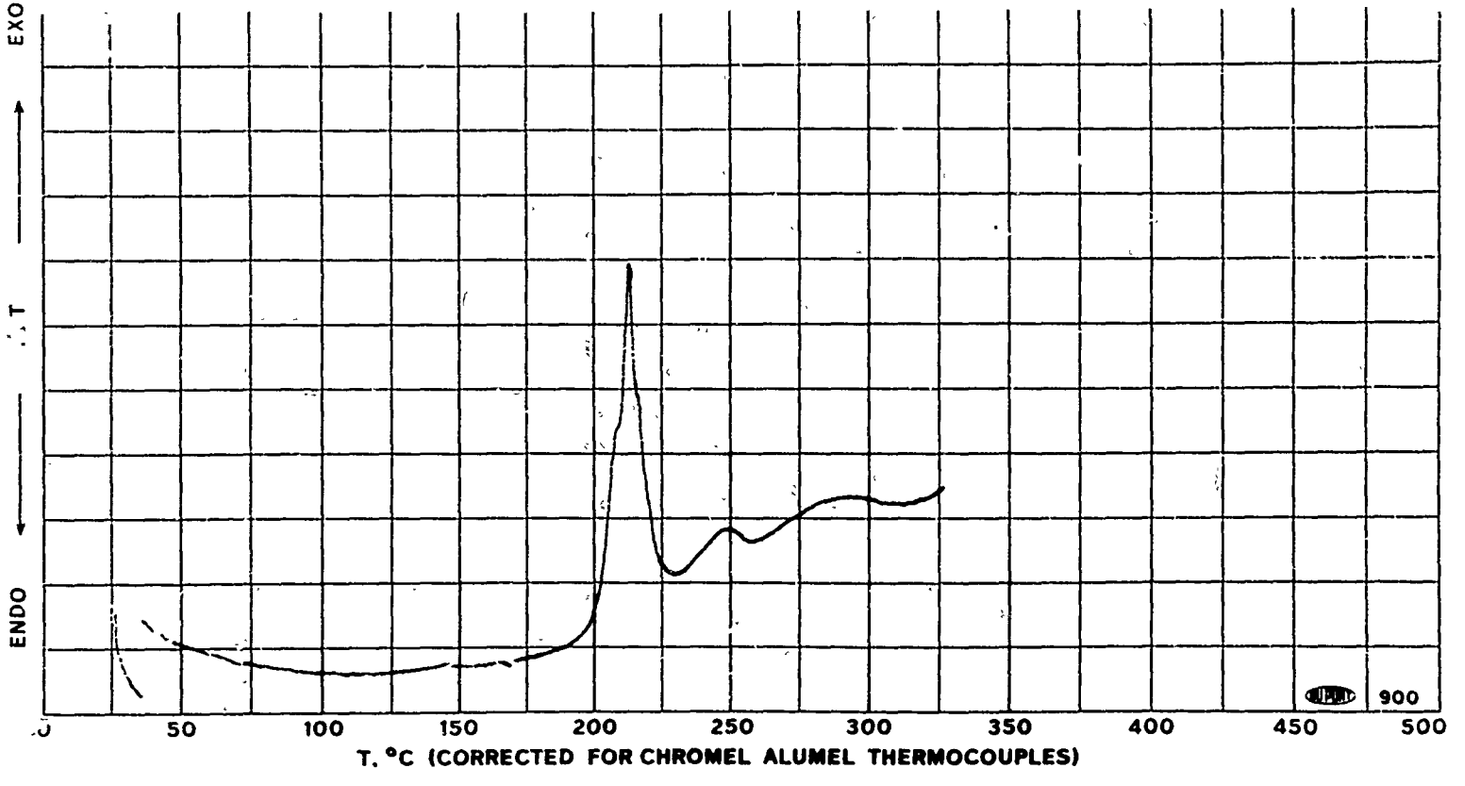


Fig A-7 DTA of KDNBF, pure

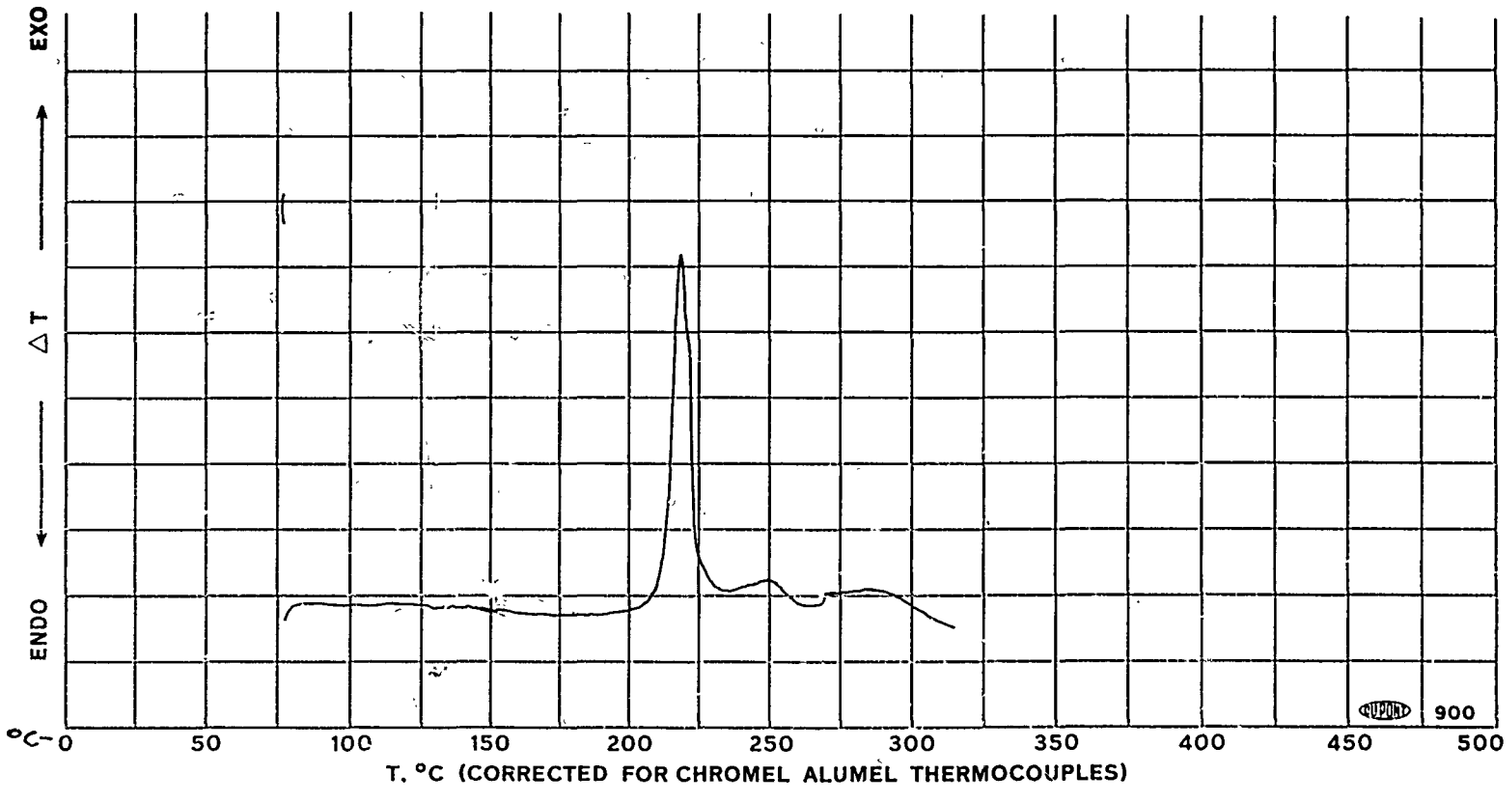


Fig A-8 DTA of KDNBF, commercial

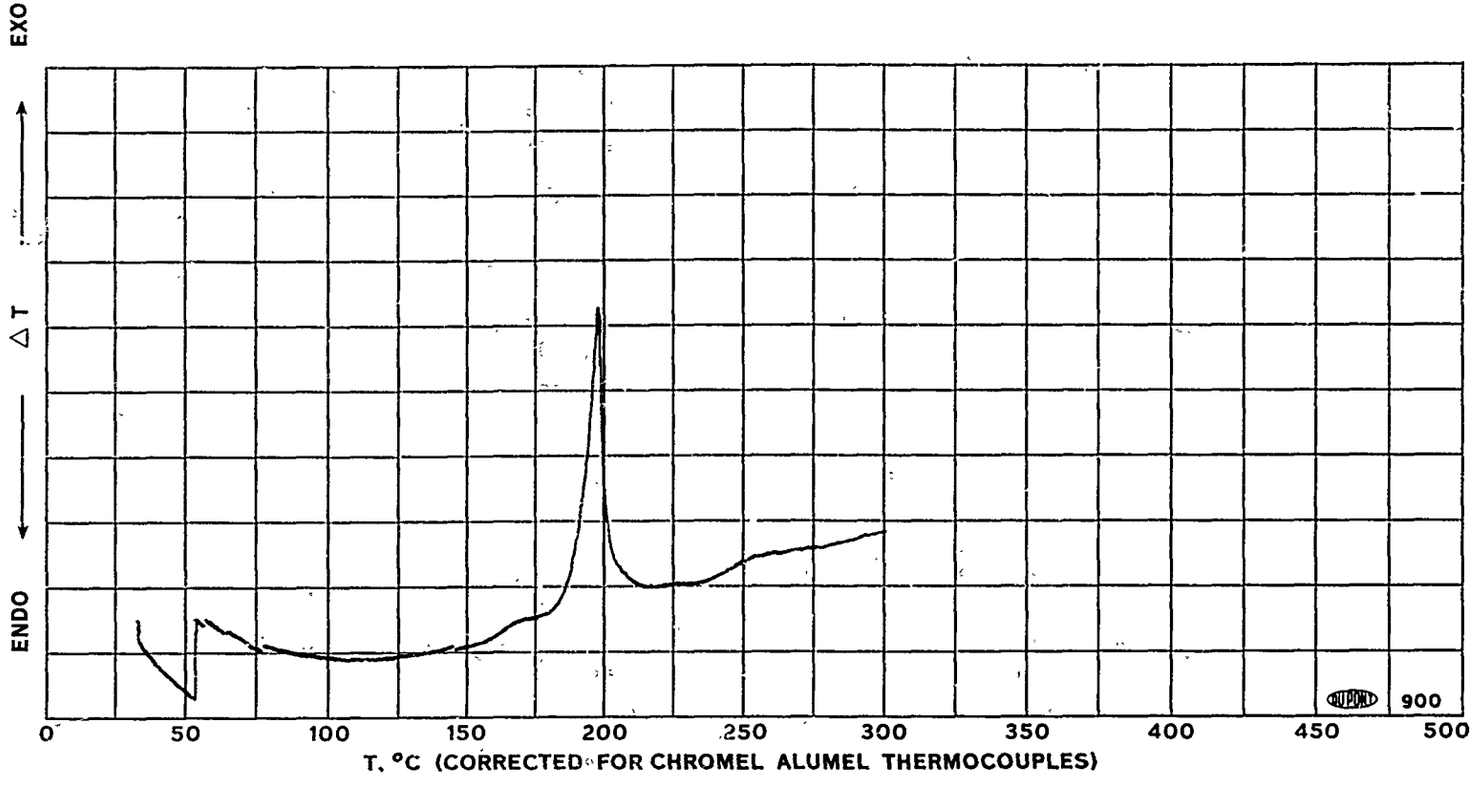


Fig A-9 DTA of KDNBF, crude (unacceptable)

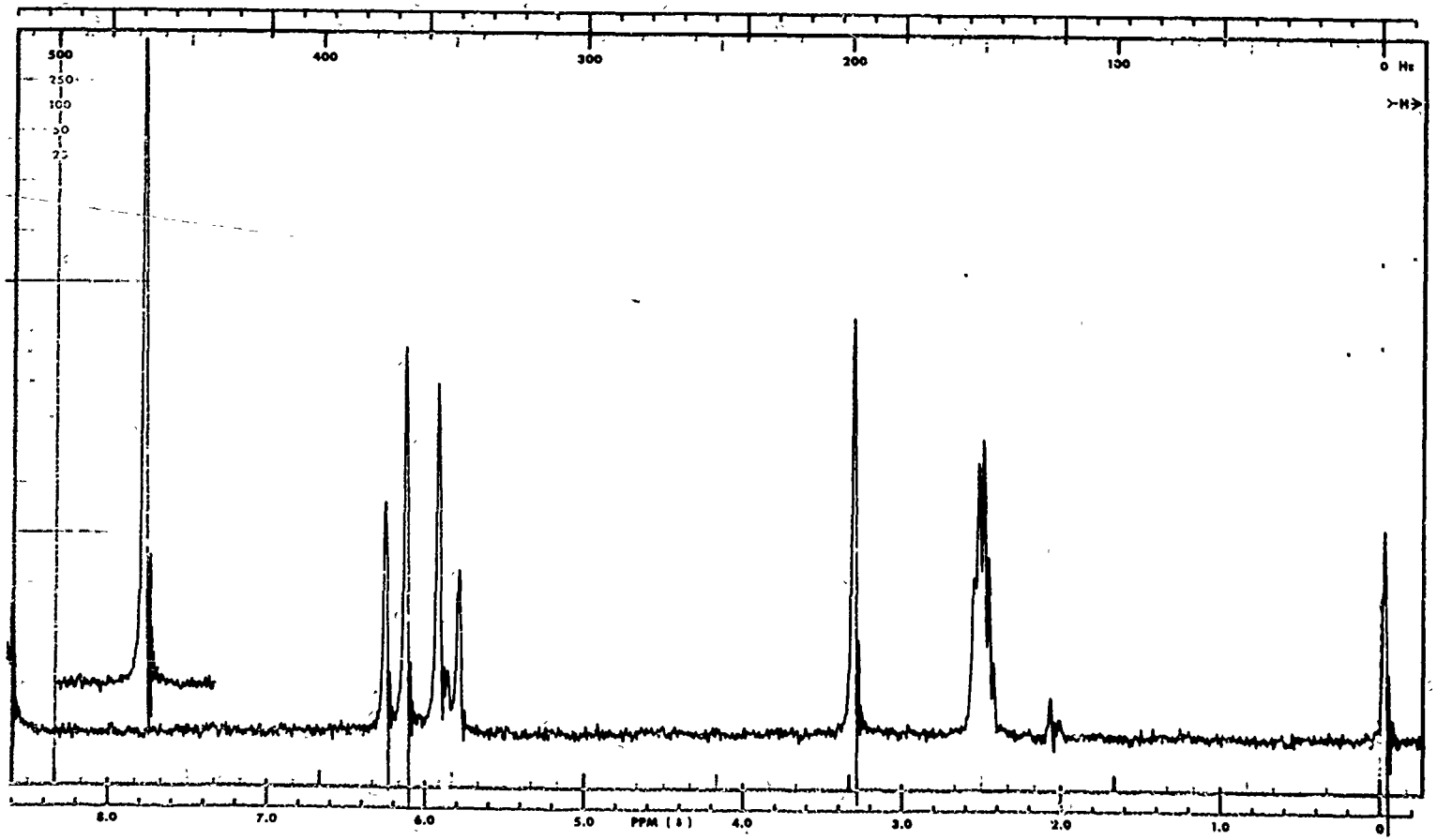


Fig A-10 NMR of KDNBF, pure

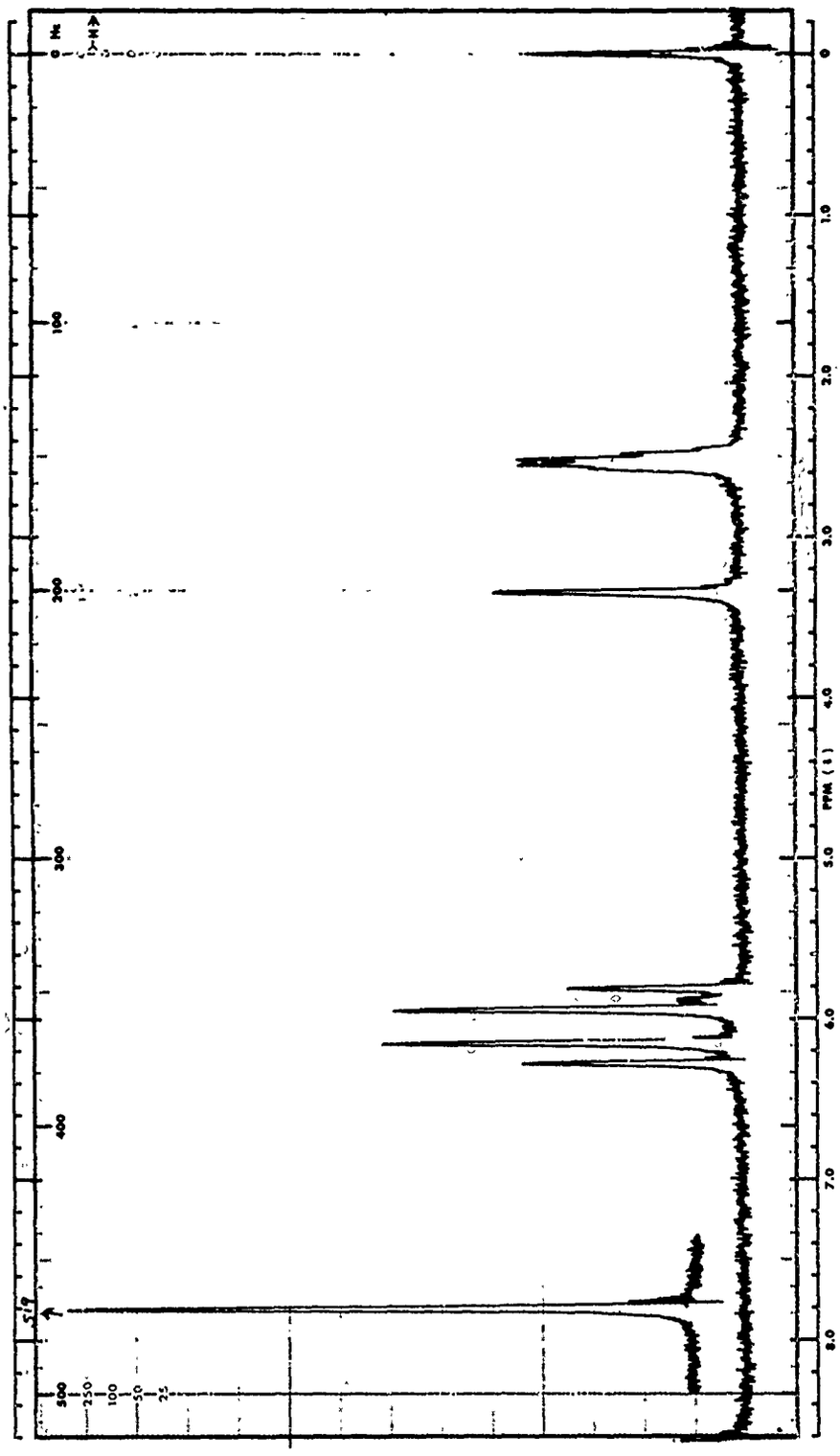


Fig A-11, NMR of KDNBF, commercial

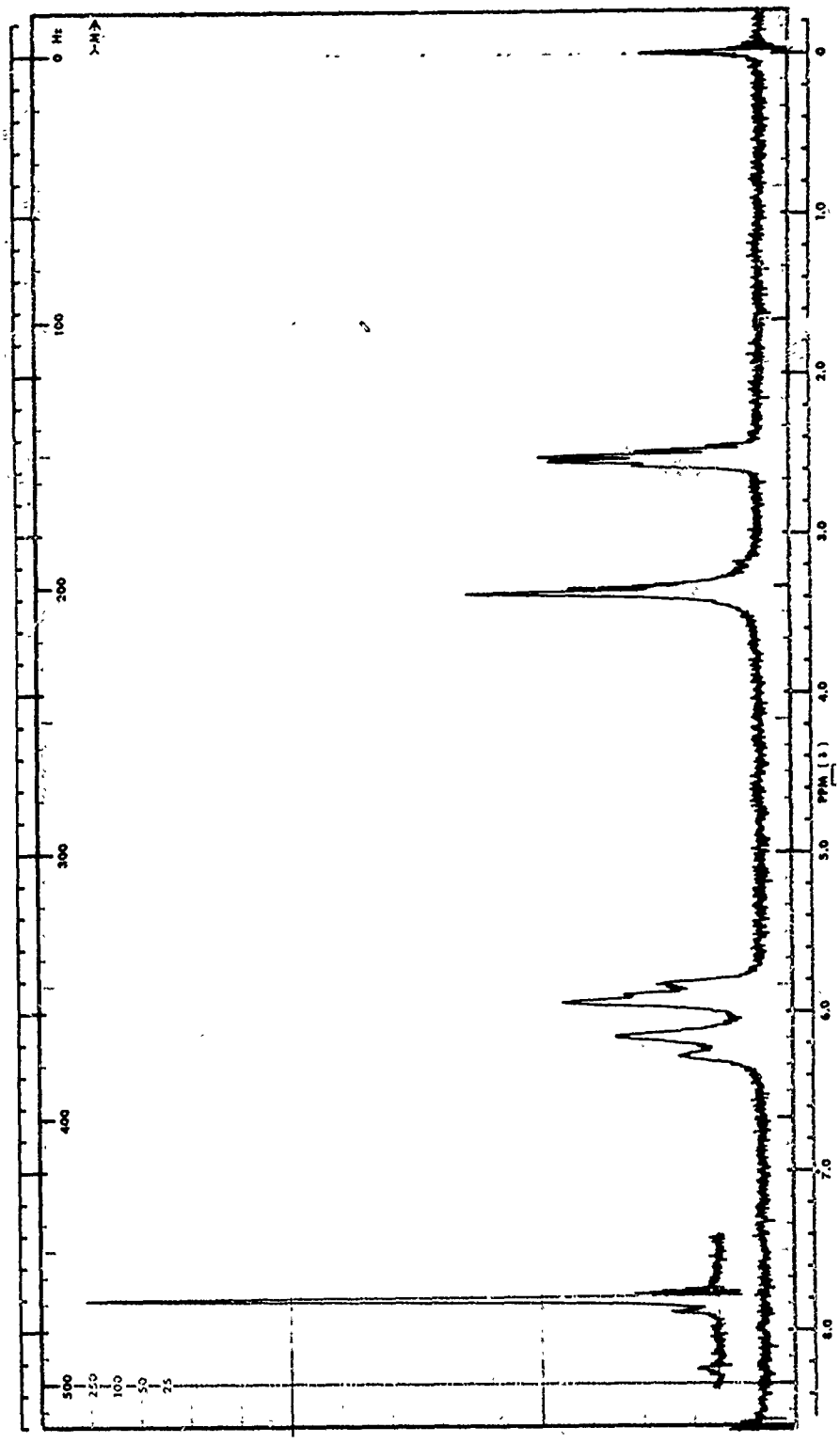


Fig A-12 NMR of KDNBF, crude (unacceptable)

NOT REPRODUCIBLE

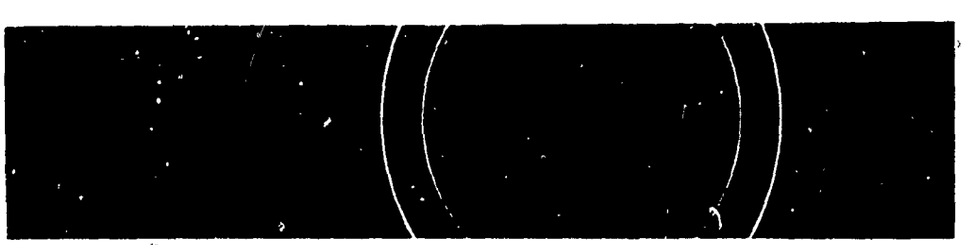


Fig A-13 X-ray diffraction pattern of KDNBF

APPENDIX B

SMUPA-VE — Proposal for the Development of a Specification for KDNBF

Chief, S/NXED, NED
ATTN: Mr. S. Roorda

Chief, Explosives Laboratory, FRL

16 June 1969
T. Costain/aeb/2812

The subject proposal is submitted for your consideration.

1 Incl
Subject Proposal
(in dupe)

RAYMOND F. WALKER
Chief, Explosives Laboratory FRL

Proposal for the Development of a Specification for KDNBF

Background

Potassium dinitrobenzofuroxan (KDNBF) has found use in various explosives actuated devices. Its desirable properties include easy ignitability, low "Z" and fast response time.

The composition was first prepared in 1899 by von P. Prost. Recently its structure was defined by R. J. Gaughran, et al, (ref. 1) and some of its characteristics determined by A. Anzalone, et al, (ref. 2). The Radiation Effects Branch of the Explosives Laboratory is presently conducting a transient radiation effects study of KDNBF, the results of which might provide data of value in connection with the preparation of a specification for this explosive.

Up till now KDNBF has been procured as a component of commercial devices with no Government specification to define the properties of the material itself. The increased use of KDNBF has generated a need for a comprehensive specification to insure that devices obtained in future procurements contain uniformly high quality KDNBF.

Purpose

It is proposed that theExplosives Laboratory conduct a program of characterization and evaluation of KDNBF to provide the data necessary for the preparation of a meaningful specification covering KDNBF. The objective will not be to grind out the usual superficial values for standard tests, but rather to apply the techniques which promise the best chance of providing direct correlation between test results and functioning in end items.

Technical Capability

The staff of the Explosives Laboratory has conducted similar programs of explosives characterization in the past. It is presently engaged in a program of evaluation of a select group of initiator compositions of British origin. The evaluation of KDNBF could be phased into the existing program with resulting economies. Those phases of the program carried out at other Arsenal segments would be under direct personal supervision of the principal investigator. Another

advantage to be considered is the complete impartiality of the Arsenal's personnel.

Technical Approach

There is a substantial supply of KDNBF stored at the Arsenal. This supply would constitute the base or normal quality material. In addition, parallel tests will be conducted on specially purified KDNBF and material simulating carelessly made or impure KDNBF. The purpose of this procedure is to establish tolerances as well as normal values. Finally the critical intermediates used in the manufacture of KDNBF would be examined (namely benzofuroxan and dinitrobenzofuroxan) and meaningful tests established to define their properties. This last is meant to circumvent the standard policy of not specifying the process of manufacture in Government specifications. Control of the properties of a material by controlling the manufacturing process is a desirable policy and one we advocate particularly for the British developed initiators mentioned previously. However, it will be necessary to change the whole philosophy of competitive bidding before such a change can be effected.

It will be necessary to supplement and affirm values previously obtained for KDNBF in such standardized tests as autoignition temperature, vacuum stability and the ordinary methods of chemical analysis. These tests are necessary for compliance with existing regulations but are not discriminating enough by themselves. Only the minimum necessary will be performed.

Greater emphasis will be placed on such techniques as differential thermal analysis (DTA), thermogravimetric analysis (TGA), ultra violet spectrophotometry (UV) and x-ray diffraction to detect subtle differences that can be correlated with the performance of the KDNFB.

The final phase of the testing process will be performance testing. Testing in simulated end items would be the most direct approach but is a destructive form of testing, the results of which would be hard to translate to other devices. A more generalized form of test would involve the use of firings in a small pressure bomb. The time vs. pressure traces as well as bridge-wire input should yield data characteristic of the KDNBF used.

Not all the data generated would be used - only that which is useful in defining KDNBF and establishing permissible tolerances.

Estimated Cost

Safety and Stability Tests

Physical Properties and Chemical Analysis

Specialized Analytical Techniques

Performance Testings

Batch Preparation and Overall Supervision by Principal Investigator

Materials and Equipment

Time Phasing

The work would be commenced as early as July 1969 and is estimated to require four months for completion.

Reporting

A progress report would be issued after 80 days and the final report would contain all raw data and our analysis and recommendations. As an addendum to the final report a purchase description for KDNBF would be prepared. This purchase description would contain all the necessary information required for the preparation of a Military Specification.

References

1. R. J. Gaughran, J. P. Picard, J. V. R. Kaufman, Contribution to the Chemistry of Benzofuroxan Derivatives J. A. C. S. 76, 2233 (1954).

2. A. Anzalone, J. Abel, A. Forsyth, Characteristics of Explosives Substances, PATR 2179, May 1955.

APPENDIX C

SMUPA-TN4 Development of a Specification for KDNBF

Chief, Explosive Lab, FRL Chief, PEL 28 July 69
WGKautz/blt/5608

1. Reference: DF from the Chief, Explosive Lab, FRL, dated 16 June 1969, subject: Proposal for the Development of a Specification for KDNBF.

2. Funding has been authorized for your lab to proceed according to the inclosed Scope of Work, Development of a Specification for KDNBF.

1 incl
Scope of Work (in dupe)

EDWIN M. ROOF
Chief, Production Engineering Lab
S/NXED

SCOPE OF WORK

DEVELOPMENT OF A SPECIFICATION FOR KDNBF

I. General Objectives

The Explosives Laboratory, FRL, will perform a four month effort for support of a program of characterization of potassium 4, 6-dinitrobenzofuroxan composition henceforth referred to as KDNBF that will define and establish permissible tolerances. Data generated will be used for the needed preparation of a comprehensive specification which will define the chemical and the physical properties of KDNBF so that a newly manufactured material conforming to the specification can be determined to be of high quality KDNBF for use in various explosive actuated devices. This material specification will be utilized by the government to assure the quality standards of the purchased product in order to prevent the use of inferior KDNBF.

The objectives of this Scope of Work will be directed to emphasize the minimum required techniques which provides direct correlation between acceptance inspection test results and functioning items, i.e., Safeguard squib switches.

II. Specific Objectives

a. Use the latest analysis techniques available which can detect the most subtle differences in KDNBF which affect the required functioning requirements.

b. Define required purity KDNBF, and methodology for its establishment.

c. Specify the process of KDNBF manufacture.

d. Conduct compatibility/stability/predictability tests.

e. Prepare loading/storage/handling procedures.

f. Prepare a Military Material Specification for KDNBF with QAD.

g. Prepare a Purchase Description for Safeguard needs.

III. Requirements

To attain the Safeguard objectives, the Explosive Laboratory shall perform the tasks as described in this Scope and as may be amended. Schedules are mandatory.

Both the tolerance and accuracy of each measurement will be recorded. The instrument(s) used and the specific test procedures shall also be recorded.

Although there is a substantial supply of KDNBF stored at Picatinny Arsenal, this supply will not necessarily constitute the basic/normal quality material. It is therefore desirable to select batches of freshly manufactured KDNBF from as many different suppliers, e.g., Atlas Chemical Company, Del Mar Engineering Laboratories, Unidynamics and Picatinny Arsenal, and assemble the KDNBF into suitable primers. These primers are to be tested under controlled conditions to determine their comparative functioning characteristics as developed by laboratory tests. The performance testing will not be left for the final phase of this project.

Performance testing will be conducted initially or concurrently with other tests in this Scope. Specifically, performance testing and simulated end items (Safeguard squib switches), a switch simulator will provide valuable information needed for evaluation of effort. Loading/handling procedures will be specified for such items. Also a more generalized form of test may make use of a small pressure bomb. The time versus pressure traces as well as bridge wire input should yield data characteristics of the KDNBF used. A more detailed study of evolution of gas versus temperature in the vacuum stability test is particularly needed.

In addition, parallel tests will be conducted on specifically purified KDNBF and the material simulating carlessly made or impure KDNBF. Purpose of this procedure is to establish tolerances as well as normal values.

In order that no unwarranted assumptions will be made and that the program will be placed on firm ground and valid conclusions can be drawn, the Explosives Laboratory will supplement and affirm values previously obtained for KDNBF by standardized tests and by the latest available discriminating techniques as well. Greater emphasis

will be placed on such techniques as differential thermal analysis (DTA), thermal gravimetric analysis (TGA), ultra-violet spectrophotometry (UV), X-ray diffraction, and other similar techniques which can be utilized to detect subtle differences that can be correlated with the performance of the KDNBF.

Included in the Military Material Specification as an addendum will be a complete Process of Manufacture of KDNBF. By controlling the manufacture process, it will be possible to control the properties of the procured material. Since literature indicates the existence of alternate reaction sequences of getting the same end product, the Explosives Laboratory will investigate various batches of KDNBF made by these different processes to determine any effects on the material characteristics on the various end products before specifying a process. Economical considerations as well as the use of the latest and most exacting procedures, equipment, etc., will be considered. If advisable, the Process of Manufacture will specify certified reagents to be used for the Materials List.

To help specify the processes and the reaction sequence of manufacture of KDNBF, critical intermediates e.g. benzofuroxan and dinitrobenzofuroxan, etc., used in the manufacture of KDNBF will be examined and meaningful tests established subject to this laboratory of S/NXED approval to define their properties.

After a Process of Manufacture is specified and KDNBF is sufficiently defined, compatibility tests with the binders, delay mixtures and surrounding metals planned for Safeguard use are needed. These tests will reflect any interfacial reactions, the rate, and degree of reaction, e.g., diffusion rate. Data on the predictability and the stability of KDNBF is required. The effects of aging (e.g. 5 years) will include the rate of degradation when exposed to different environments. Differences between old and new KDNBF, no matter how subtle, especially performance characteristics, will be reported. Results of all these tests will reflect the degree of concern which needs to be observed in regard to detrimental effects on the performance of the end item. Storage conditions will be specified so as to retard or at least minimize any of these effects.

IV. Time Phasing/Funding/Reporting

This Scope of Work, Development of a Specification for KDNBF will commence 1 Aug 69. Funding documentation has been initiated for a period of 1 Aug 69 thru 30 Nov 69 to the amount of _____ for the effort specified in this Scope. Funds are available on _____ .

A Monthly Narrative Progress Report is required by DF to this office on or before the first day of the month. The first report will be due by 1 Sep 69. Funding reports will be in accordance with existing Arsenal Regulations. In addition, a project report which includes all items required by this Scope , analyses, and recommendations which will be received by this office no later than 30 days after this project's projected completion date, 30 Nov 69.

FRL personnel involved are expected to work closely with Production Engineering Laboratory, S/NXED, engineers in all aspects of the project. Messr. A. Scigliano and W. Kautz, ext. 5608, are the Project Officers on this task.

UNCLASSIFIED

Security Classification

DOCUMENT CONTROL DATA - R & D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

1 ORIGINATING ACTIVITY (Corporate author)	2a. REPORT SECURITY CLASSIFICATION
Picatinny Arsenal, Dover, New Jersey	UNCLASSIFIED
	2b. GROUP

3. REPORT TITLE

INVESTIGATION OF POTASSIUM DINITRO-BENZOFUROXAN (KDNBF) TO PROVIDE DATA NECESSARY FOR THE PREPARATION OF A MILITARY SPECIFICATION

4. DESCRIPTIVE NOTES (Type of report and inclusive dates)

5. AUTHOR(S) (First name, middle initial, last name)

T. S. Costain

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8a. CONTRACT OR GRANT NO.	9a. ORIGINATOR'S REPORT NUMBER(S)
b. PROJECT NO.	Technical Report 4067
c. AMCMS Code 4280.26.90001.6	9b. OTHER REPORT NO(S) (Any other numbers that may be assigned this report)
d.	

10. DISTRIBUTION STATEMENT Each transmittal of this document outside the agencies of the U. S. Government must have prior approval of the Scientific and Technical Information Branch, Picatinny Arsenal, Dover, New Jersey.

11. SUPPLEMENTARY NOTES	12. SPONSORING MILITARY ACTIVITY

13. ABSTRACT

Under a program funded by the Nuclear Engineering Directorate, the Explosives Laboratory of Feltman Research Laboratories, Picatinny Arsenal, has defined the critical properties of KDNBF (commonly known as potassium dinitrobenzo-furoxan, but more accurately designated as potassium 4, 6 dinitro- 7 hydroxy-7 hydro-benzofuroxan), established a self-checking testing procedure, and set tolerances which can be used as a basis for a rigorous military specification. KDNBF's of both particularly high and low purity and quality were tested, concurrently with KDNBF's obtained from three commercial sources to provide a broad diversity of data. The functioning of KDNBF was defined from pressure-time traces obtained from firings in a 1-ml (milliliter) pressure bomb, and it was related to purity as determined by nuclear magnetic resonance, X-ray diffraction, and elemental analysis. The thermal stability, as measured by differential thermal analysis and gas evolution at 120°C, also was related to KDNBF purity and function. A specific test based on solubility in methanol was devised to detect contamination of KDNBF by a hydrolysis decomposition product and, in addition, storage under absolute methanol in sealed containers was recommended. Of secondary importance were tests related to color, form, granulation, and bulk density, which were used mainly to describe a form of KDNBF suitable for use in automatic-loading machinery. In the course of the study the manufacturing parameters for KDNBF were systematically varied so that a judgment on the effects and relative importance of raw materials, intermediate compounds, and unit processes could be reached. A recommended process of manufacture is presented.

DD FORM 1 NOV 65 1473 REPLACES DD FORM 1473, 1 JAN 64, WHICH IS OBSOLETE FOR ARMY USE.

UNCLASSIFIED

Security Classification

14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Critical parameters of KDNBF						
One milliliter pressure bomb						
Pressure-time traces						
Nuclear magnetic resonance						
X-ray diffraction						
Ultraviolet and infrared spectroscopy						
Elemental analysis						
Differential thermal analysis						
Gas evolution at 120°C						
Impact-sensitivity test						
Explosion-temperature test						
Vacuum stability test						
Bulk density						
Sieve analysis						
Hand-loading grade						
Automatic-loading grade						
Contamination by methanol-soluble substances						